Blast Furnace Burden Softening and Melting Phenomena: Part III. Melt Onset and Initial Microstructural Transformations in Pellets

PAULO F. NOGUEIRA and RICHARD J. FRUEHAN

The softening and melting of the ferrous burden in the blast furnace occur in the cohesive zone, affecting the furnace's performance. This work was designed to better understand these phenomena. In this article, the onset of melting and the evolution of the microstructure before softening were analyzed using a confocal scanning laser microscope (CSLM). Experiments were performed with industrial or synthetic pellets prereduced to wustite, placed over a metallic iron liner, and heated at either 1 °C/min or 3 °C/min under argon. The samples showed exudation of liquid, which coated the liner at temperatures above 1150 °C. The melt onset for FeO-SiO₂ synthetic samples occurred at similar temperatures, regardless of the experimental conditions. The melt onset temperature for the basic samples was lower than the acid samples. A sudden increase of the amount of liquid occurred first for the acid samples. A transition in the microstructure with wustite particles permeated by an intergranular (slag) phase was observed in the industrial samples. These results suggest that the initial liquid does not directly influence softening; rather, it acts to improve mass transport and to push the system to a semisolid.

I. INTRODUCTION

EVEN though direct reduced iron (DRI) production increased in the past decade and smelting processes are maturing, the blast furnace is still the predominant process for the reduction of iron ore. The cohesive zone has a significant effect on the efficiency and productivity of the furnace. In this region, the metallic burden softens, drips liquid, and melts. In the meanwhile, the metallic burden loses permeability, forcing the gas flow through the coke layers. The deformation of the ferrous burden unit is the macroscopic result of softening. As the temperature increases, the liquid fraction increases until the burden unit cannot hold, resulting in the dripping of liquid. The final step is the final meltdown. This article is part of a series aimed at studying the underlying phenomena that occur in the cohesive zone. Two previous articles presented the observation of the interaction between bulk pellets during softening and melting^[1] and the microstructural transformations after softening and before melting.^[2] In this article, the importance of the presence of liquid in the early stages of softening will be discussed, as well as its implications in the transformations of the microstructure that are involved in the early stages of softening.

In the present article, these observations are complemented with those performed with the confocal scanning laser microscope (CSLM) at lower temperature. To validate some of these observations, experiments were performed using synthetic FeO-SiO₂ pellets. These experiments were

Manuscript submitted July 14, 2004.

designed to provide a better understanding of the phenomena occurring at the microscopical level that leads to the formation of a significant amount of liquid in the pellet. As demonstrated by Bakker,^[3] the onset of liquid is the key factor leading to softening. The initial oxide liquid will form at the interface of lowest melting point, usually between an iron oxide particle and another oxide. It depends on the phases present and their distribution. After the first melt is formed, it will tend to wet the ore particles due to the reduction of the interfacial energy.^[3] This rate of liquid spreading would depend on the viscosity of the slag formed. Furthermore, as the liquid spreads, it may encounter other oxide particles and would react with them if it lowers the system energy, and thus pushes the system toward equilibrium. The burden unit would become a semisolid material when the liquid phase covers the oxide particles, acting as a lubricant.^[3] Thus, after the liquid slag in the core has wet all the oxide particles, the core has a very reduced mechanical strength and the resistance to deformation will be determined by the iron shell.^[3]

II. LITERATURE REVIEW

A thorough literature review on softening and melting is presented in part I of this series.^[1] In this article, discussion will be limited to the phenomena surrounding the melt onset. The loci for melt onset are the low melting point ones, generally the interface between two particles, which is usually surrounded by a slag film in pellets. The common gangue materials in the iron ore are silica and alumina. The addition of flux was initially done to enhance slag formation, reduce the fine amount, and recycle waste materials. In some cases, they also happened to improve the softening and melting properties. Currently, pellets and sinter are produced with flux additions, usually bearers of CaO and MgO.

The slag forming oxides influence the melt onset through different phenomena. The softening temperature, which is

PAULO F. NOGUEIRA, formerly Graduate Student with the Center for Iron and Steelmaking Research, Department of Materials Science and Engineering, Carnegie Mellon University, is Senior Researcher, with Ferrous Products R&D, Cia. Vale do Rio Doce CVRD, Vitoria, ES, 29090-900 Brazil. Contact e-mail: paulo.freitas.nogueira@cvrd.com.br RICHARD J. FRUEHAN, Professor, is with the Center for Iron and Steelmaking Research, Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA 15213.

related to liquid onset, increases with the reduction of the dolomite particle size, according to Bakker^[3] and Barnaba,^[4] demonstrating that a fast dissolution of the flux agents into the slag improves the overall properties. Khaki et al.^[7] showed that the increase in the fraction of slag formers reduces the dripping temperature. The dependency of the softening and meltdown temperatures with basicity was studied by Barnaba,^[4] Khaki *et al.*,^[5] and Ritz *et al.*,^[6] experimentally. They found that both temperatures increase with basicity. It was observed experimentally that CaO substitutes for FeO as the basicity increases.^[2] This affects the viscosity, as discussed in part 2 of this series.^[2] A beneficial effect of MgO is that it increases the viscosity of the slag at 1450 °C, but reduces it at higher temperatures, so that the slag drips together with the metal.^[4] According to Bentell.^[7] the addition of MgO in lieu of CaO is beneficial, because it will not form low melting point slags, especially at high MgO/SiO₂ ratios. Also, at high ratios, it is possible that the FeO matrix will melt earlier than the slag.^[7]

The influence of the reduction degree on the softening and melting phenomena occurs by two different mechanisms. The variation of the reduction degree will determine the volumetric fraction of metallic iron. Whereas the iron shell can hold the liquid slag, the dripping temperature may depend on its strength, which is a function of the iron layer thickness.^[1,3,4] Another effect of the reduction degree occurs by reducing the amount of FeO that can act as a slag former.^[5,8,9] In both cases, the higher the reduction degree, the higher should be the dripping start temperature. Several factors influence the reducibility of the burden material, which controls the reduction degree in the furnace. The addition of fluxing agents tends to increase the reducibility of the burden materials.^[10] This may be the reason why Borider^[8,9] found that the metallic iron content had an effect on the softening and melting temperatures of both olivine fluxed and acid pellets, but not on the basic pellets.

III. EXPERIMENTAL PROCEDURE

The observation of the melt onset was done in the CSLM. A description of this equipment has been presented previously.^[11] In this article, three samples were used: acid, basic, and synthetic FeO-SiO₂ pellets. The compositions of these pellets are presented in Table I. The synthetic pellets

 Table I.
 Composition, Basicity, and Amount of Slag Formers of the Pellets Used in this Work

Component	Basic	Acid	Synthetic
Fe ₂ O ₃	90.09	92	90
FeO	0.39	0.39	_
SiO ₂	4.01	4.91	10
Al ₂ O ₃	0.43	0.31	_
CaO	4.22	1.14	_
MgO	0.99	0.56	_
MnO	0.26	0.19	_
P_2O_5	0.092	0.105	_
Na ₂ O	0.043	0.017	_
K ₂ Õ	0.03	0.03	_
Basicity	1.052	0.232	_
Expanded basicity	1.299	0.346	_
Slag formers (pct)	10.08	7.27	10

were prepared by mixing reaction grade silica and hematite, drying, and then firing in air at 1200 °C for 2 hours. These samples were prereduced to wustite using a CO/CO_2 equimolar mixture at 800 °C, following the procedure described previously.^[1] Some pellets of the synthetic batch were also heat treated at 800 °C under CO/CO_2 for 5 hours.

After prereduction, these samples were ground to 1200 grit SiC grinding paper and cut to cross sections of about 1 mm or less thickness. These sections were then cut to fit a 10-mm CSLM alumina crucible, next to a B-type thermocouple. These were calibrated *in situ* against the melting point of copper and nickel. The measured temperature was then corrected assuming the variation between both melting points to be linear. A correction to account for different emmisivities was done by measuring the melting point of pure wustite in equilibrium with iron.^[12] The results were then corrected linearly by 56 °C for the first series and 47 °C for the second.

The samples were placed in the CSLM chamber, which was evacuated for 15 minutes. The heating was done under argon flow, previously purified by passing through Cu turnings at 400 °C. The samples were initially heated to 950 °C and, after a 5-minute holding period, were further heated at a rate of either 1 °C/min or 3 °C/min. When the desired temperature was attained, the power was shut down and the samples cooled at a rate of approximately 800 °C/min to about 300 °C. After cooling, the surface of these samples was observed in the scanning electron microscope (SEM), and then they were mounted and polished for SEM-energydispersive x-ray analysis (EDX). The movies obtained after the experiments were analyzed to determine the melt onset temperature (the first temperature where the presence of liquid is clear). Figure 1 presents stills of one of the movies of the surface of a synthetic pellet, prereduced to FeO and heat treated, being heated to 1247 °C at 3 °C/min. This figure shows the appearance of liquid at 1187 °C.

IV. EXPERIMENTAL RESULTS

A. Synthetic Pellets

After the preliminary experiments in the CSLM,^[13] it was questioned whether the microstructural transformations were controlled by kinetics or determined by thermodynamic equilibrium. Therefore, a simpler system was chosen, the FeO-SiO₂, with a well-determined phase diagram.^[14] Two distinct series were performed, one with the as-prereduced pellets and another with the heat-treated samples. The main difference between both is the presence of coarse silica particles in the heat-treated samples and of slag peppered with what seem to be silica precipitates in the "aspre-reduced" samples. The liquid onset in the as-pre-reduced samples was observed between 1167 °C and 1171 °C in the experiment heated at 1 °C/min. A similar result was obtained in the experiment performed with a heating rate of 3 °C/min. The liquid onset temperature in the heat-treated samples was also reasonably close to the samples heated at either 1 °C or 3 °C/min. However, the temperature was approximately 10 °C higher than the untreated samples. In the sample heated at 1 °C/min, the liquid began to appear at 1182 °C, while in the sample heated at 3 °C/min, it appeared between 1177 °C and 1187 °C. The better surface



Fig. 1—Snapshots of the video of the surface of a synthetic (FeO-SiO₂) pellet, prereduced to "FeO" and heat treated, while being heated to 1247 °C at 3 °C/min.

quality of the sample used in the experiment heated at 3 °C/min and shown in Figure 1 complicated the initial observation of liquid. It only becomes clear above 1187 °C. In this figure, it is also possible to note the evolution of the microstructure even below the melt onset temperature.

The microstructure of these samples evolved from the original microstructure to a slag matrix permeating wustite islands one, as shown in Figure 2. Although the initial microstructures of both "heat-treated" and "as-reduced" pellets were different, a similar result was achieved at temperatures higher than 1247 °C. Figure 3 shows an example of an as-reduced sample, and Figure 4 shows one of a heattreated sample, in this case, both heated at 3 °C/min to 1147 °C. The samples quenched from 1147 °C, heated at 1 °C/min, appear similar to those obtained from the aspre-reduced sample. Besides this case, the microstructure of all the remaining samples appears the same within a given condition. These show a 100 °C interval where the microstructure changes from the "as-received" to the solidliquid equilibrium. They also suggest that the equilibrium structure at higher temperatures is that of the samples after sintering.

The slag phase from the quenched samples is actually composed of two phases, as can be seen in these figures. The darker phase, usually with a dendritic or lamellar aspect, has a higher SiO_2 content than the lighter phase occupying the interdendritic regions. It is interesting to note that despite the initial composition of the slag and of any event during the heatup, the result was similar for both pellets. The dark and light gray phases have indistinguish-



Fig. 2—Polished section of synthetic pellet prereduced to wustite and heated to 1247 °C at a heating rate of 1 °C/min, showing the presence of wustite and the two components of the intergranular "slag" phase (SEM-backscattered electron image (BEI)).

able compositions when compared with their peers (different heating rate or pretreatment). The composition and morphology of the dark gray phase indicate that it should be fayalite (2FeO·SiO₂), approximately 30 wt pct SiO₂, while the actual slag composition would then be that of the wustite-fayalite eutectic (24 wt pct SiO₂). The discrepancy between the values obtained analytically and those from the phase diagram is relatively small (~2 to 5 pct) and can be due to a combination of factors such as the imprecision of



Fig. 3—Polished section of a synthetic pellet prereduced to wustite and heated to 1147 °C at a heating rate of 1 °C/min, showing the presence of the intergranular slag phase, the dark particle clusters, and the wustite particles (SEM-BEI).



Fig. 4—Polished cross section of a synthetic pellet prereduced to wustite, heat treated at 800 °C, and heated to 1147 °C at a heating rate of 3 °C/min, showing the presence of the wustite and silica particles, as well as the intergranular slag phase (SEM-BEI).

the technique, effect of the gold coating, high fast cooling rate resulting in the entrapment of silica in those phases, among others.

B. Acid Pellets

The stills of the video obtained in one of the experiments taken to 1256 °C at a heating rate of 3 °C/min show the disappearance of a gangue particle between 1056 °C and 1066 °C. Despite the disappearance of this particle, the amount of liquid slag only was sufficient to be observed at 1146 °C. Similarly, in the experiment heated at 1 °C/min, the fraction of liquid rose significantly between 1156 °C and 1206 °C.

The microstructure of the samples heated until 947 °C over 5 minutes and quenched after a holding period of 5 minutes is similar to that of the as-pre-reduced sample. This result indicates that although there appears to be liquid evolution in the CSLM at this temperature, there is no significant transformation in the microstructure of the sample. This is not the case in the samples quenched from

554-VOLUME 37B, AUGUST 2006

1047 °C, heated at either 1 °C or 3 °C/min. Although the microstructures of these samples are similar among themselves, a spreading of the slag phase can be seen. Upon closer examination, it can be noted that the volume fraction of slag is increasing, in agreement with what was seen in the CSLM. In both samples, the distribution of the slag phase is heterogeneous, and slag pockets are located probably near the original gangue particle. From qualitative elemental EDS maps, it was noted that in the majority of the samples, the second most prevalent oxide in the oxide solid solution, after FeO, is silica (circa 35 pct).

In the samples heated to 1047 °C, the difficulty in differentiating the two heating rates indicates that this parameter does not appear to be relevant. It is interesting to note that the polished image shows an intermediate stage between that sample and the following samples. The fraction of slag phase increased significantly, although it was still confined to certain regions of the sample, contrary to what was observed in the samples quenched from 1147 °C and 1247 °C, where a slag matrix wustite islands microstructure was observed. In the samples heated at 1 °C/min, the FeO islands are coarser than those in the samples heated at 3 °C/min. In addition, the islands in the samples quenched from 1247 °C are coarser than those in the samples quenched from 1147 °C. Both are due to longer periods at a higher temperature endured by those heated at 1 °C/min or higher temperatures. The Si $K_{\alpha 1}$ elemental maps from the samples quenched from 1047 °C and 1097 °C at both heating rates showed this transition in better detail, as can be seen in Figure 5. The samples quenched from 1047 °C still show the massive presence of gangue particles, with varying sizes and shapes, similar to the as-reduced pellet. In the sample quenched from 1097 °C after a heating rate of 1 °C/min, it can be seen that there are no gangue or flux particles, only a Si-rich area which is combined with Ca and Fe, indicating that it is homogenizing. On the other hand, for the sample quenched from the same temperature, but heated at 3 °C/min, it was seen that there are still gangue particles left, albeit with a smoother shape. This difference is attributed to longer residence time for the samples heated at 1 °C/min.

The slag phase of all samples presented previously was analyzed using EDX in the SEM. The results show an overall tendency of reducing the value spread, as well as increasing the FeO and reducing the SiO₂, CaO, and MgO contents of the slag. These results suggest that as the temperature rises, the dissolution of the gangue particles decreases the basicity to levels close to those predicted by the bulk composition.^[1] Furthermore, the reduction in the analyzed spread at each temperature indicates that the sample is homogenizing, which is corroborated by the microstructural transformations, thereby confirming the threshold in the microstructure between the temperatures of 1047 °C and 1147 °C.

C. Fluxed Pellets

The behavior of the basic pellets was somewhat similar to the acid and synthetic pellets. In the video obtained in an experiment where a basic pellet is heated to 1256 °C at 1 °C/min, it is possible to see something that appears to be the melting of a particle at 1028 °C, which then disappears



Fig. 5—Si $K_{\alpha 1}$ EDX maps of the polished surface of an acid pellet, prereduced to wustite. These maps show the residual gangue particles present at 1047 °C and dissolving into Si-rich clusters at 1097 °C.



Fig. 6—Si $K_{\alpha 1}$ and Ca $K_{\alpha 1}$ EDX maps of the polished surface of a basic pellet, prereduced to wustite. These maps show the presence of residual gangue and flux particles at 1047 °C that became Ca- and Si-rich clusters at 1147 °C.

at 1036 °C. At approximately 1086 °C, the surface of this sample becomes very faceted and, at 1147 °C, appears more signs of liquid. The sample heated at 3 °C/min had a very similar behavior. The analysis of the video showed that the first indications of liquid were observed at 1082 °C, but remained low up to 1185 °C.

As in the acid and synthetic pellets, the microstructure of this sample evolves from heterogeneous, "orelike," to homogeneous, with wustite islands and a slag matrix permeating the sample. However, this transformation occurs at temperatures slightly higher than in the acid pellet. The basic samples quenched from 1147 °C have a microstructure similar to the acid samples quenched from 1097 °C (Figure 5). In addition, the samples quenched from 1197 °C and above have the wustite islands/slag matrix structure. This indicates that there is also a threshold, and that it occurs at an interval 50 °C longer than for the acid pellets, and is corroborated by the Si $K_{\alpha 1}$ and Ca $K_{\alpha 1}$ elemental maps of the basic samples quenched from 1047 °C and 1147 °C heated at either 1 °C/min or 3 °C/min, as shown in Figure 6. The slag matrix was analyzed by SEM-EDX. The results obtained showed a trend similar to the acid pellets: a decrease in the basicity as the temperature rose. There were intact gangue and flux particles in the sample after prereduction, with more of the former. The dissolution of these particles would be sufficient for this slight decrease in the basicity. Unlike that observed with the acid pellets, the spread of the chemical composition did not decrease with the increase in temperature. The flux particles added another complicating factor to the system, and now two different particles had to dissolve for the samples to become homogeneous.

D. Liquid Exudation

The analysis of the experiments with basic pellets heated to 1256 °C showed a discrepancy between the video stills and the microstructure of the cooled sample with regard to the apparent liquid volume. Considering that the fraction of slag formers in this sample is slightly higher than 10 pct, and because the microstructure was devoid of second-phase particles, these oxides must have been incorporated into a slag. In most of the experiments performed with the metallic iron liner, the samples heated higher than approximately 1150 °C adhered to it. Figures 7 and 8 present



Fig. 7—Surface of an acid pellet sample prereduced to wustite and heated at 3 $^{\circ}C/min$ to 1147 $^{\circ}C$ (SEM-BEI).



Fig. 8—Surface of an acid pellet sample prereduced to wustite and heated at 3 °C/min to 1247 °C (SEM-BEI).

examples of acid samples heated to 1147 °C or 1247 °C, at a rate of 3 °C. These samples show that although the samples heated to approximately 1150 °C had adhered to the liner, there is no apparent exudation of liquid from the sides (Figure 7). Both samples seem to be flush with the liner underneath. The same cannot be stated for the samples heated to approximately 1250 °C. A similar behavior was encountered with the basic pellets. Although both samples have indices of a liquid covering the edge between the exposed section of the liner and the section, the basic pellets had exuded more liquid than the acid pellets. In fact, in one of these samples, it is nearly impossible to locate the wustite islands. Similar results were observed in both the acid pellets and synthetic pellets, and it was found that these results were more related to the thickness of the sample than any other variable. It appears that thicker samples better held their shape at these temperatures.

V. DISCUSSION

This new experimental technique was successful in determining the appearance of liquid. These results are presented in Table II. It is interesting to note that the values for experiments at different heating rates are within the experimental precision. This indicates that the kinetics of melting is not a major factor. In the experiments performed with synthetic pellets, the results show that the melt onset temperature was very close to the solidus from the phase diagram. In the heat-treated samples, the better quality of the surface hindered the observation of liquid in some cases, but the values were still within the expected experimental limitations. In the samples where liquid was observed, the solidification products were secondary wustite, fayalite, and a wustite-fayalite eutectic, coexisting with primary wustite. This occurred regardless of initial sample condition.

All samples heated to more than 1147 °C adhered to the metallic iron surface they laid on. It was seen that in the samples cooled from approximately 1250 °C, some of the liquid exuded from the sample and partially coated the liner. In the samples quenched from approximately 1150 °C, on the other hand, there was no sign of external exudation of liquid slag. These findings were observed for both acid and basic pellets. The better coverage observed for the basic pellet's slags in comparison with the acid's slags suggests that the difference in the work of adhesion is more negative for the basic slag than for the acid slag. In industrial situations, the pellet has a significant amount of metallic iron when it reaches the cohesive zone. As mentioned in the second article of this series,^[2] the data for surface tension at 1250 °C is almost nonexistent. Also, the difference in viscosity between the slag from the basic and acid pellets could be of an order of magnitude, which could justify the observed difference in behavior.^[2] The better wetting of the metallic iron liner by the basic slag suggests that this slag would have a stronger tendency to stay in the pellet or sinter than in the acid pellets, and therefore could be another factor explaining the difference in exudation behavior. However, these results are just indications, and experiments for the measurement of contact angles between iron and these slags should be carried out.

Table II. Melt Onset Temperatures of the Samples Tested in the CSLM

Melt Onset Temperature (°C)					
Heating Rate	Acid	Basic Fluxed	Synthetic	Synthetic Heat Treated	
1 °C/min 3 °C/min	1147 1146	1028 1082	1167 to 1172 1170	1177 to 1187 1187	

The liquid onset occurred at much lower temperatures for the complex samples (acid and basic), before any significant microstructural transformation. This is due to the fluxing effect of alkaline oxides. Although these oxides are present at less than 0.1 pct, they can reduce the solidus temperature significantly, even though the fraction of liquid at this point is small. This aspect will be discussed in detail in part IV of this series. The melt onset in the basic pellets was observed at temperatures lower than the acid pellets, while the softening and melting results indicate the contrary.^[1] In these pellets, it seemed that there was a "double rise" in the liquid fraction. In the acid pellets, at approximately 20 °C after the melt onset, a sharp increase in the fraction of liquid was observed, while for the basic pellets, this occurred between 50 °C and 100 °C after the first liquid was observed. These results indicate that this sharp rise in the fraction of liquid may be more relevant for the high-temperature properties than the first liquid onset.

In all experimental series, a similar behavior was observed, a transition from the prereduced microstructure, similar to the fired pellet microstructure, to that characteristic of a semisolid, with wustite islands in a slag matrix. The temperature at which this transition happened as well as its duration varied. It appears that only after sufficient liquid has formed does the microstructure of the system tend toward equilibrium, regardless of the initial condition. Between the temperatures of 1047 °C and 1097 °C, the gangue and flux particles in the acid pellets start to dissolve. Once the microstructure has evolved into the slag matrix with wustite island morphology, the reduction in the chemical composition spread indicates a homogenization of the liquid slag and the system tends toward equilibrium. Similar behavior was observed in the basic pellets under the same conditions, although the threshold occurs at higher temperatures between 1056 °C and 1206 °C, and the chemical composition of the slag showed a wider spread at around 1250 °C. At this temperature, it is impossible to distinguish the microstructure of any of the samples from each other.

Thus, the sample evolves from an "as-fired" microstructure to a semisolid throughout the heating. The liquid acts to improve the mass transport of the heterogeneously distributed components; thus, this evolution is controlled by the quantity of liquid present. In the samples studied in this article, the dissolution of the gangue and flux particles occurred at relatively low temperatures, eventually leading to a homogeneous structure at 1250 °C. For the slow heating rates normally encountered in the blast furnace, the threshold is independent of it and thermodynamics is the key factor in the microstructural homogenization of these pellets. The question arising from this study is how much liquid is necessary to lead the system toward equilibrium.

VI. CONCLUSIONS

In this research, a unique experimental technique (CSLM) was used to measure the melt onset in wustite pellets, of synthetic or industrial origin. However, this measurement is strongly influenced by the surface roughness. The microstructural transformations near softening were possible to examine by interrupting these experiments at different temperatures. The major findings are as follows.

- 1. The experimental technique using the CSLM was successful in seeing the melt onset in ferrous materials.
- 2. The melt onset temperature measured for the synthetic pellets with different heating rates or pretreatment was within the experimental precision of the technique for all experiments. This indicates that up to 3 °C/min, the melt onset in these samples is controlled by thermodynamics not kinetics.
- 3. All samples heated above 1147 °C adhered to the metallic liner or the crucible. The slag from the basic pellets seemed to cover a larger area of the liner.
- 4. The melt onset temperature in the basic samples was approximately 100 °C lower than the acid samples.
- 5. A sudden increase of the amount of liquid was observed close to the softening temperatures measured in laboratory experiments^[1] in both acid and basic samples. It occurred first for the acid samples.
- 6. A transition in the microstructure of the burden from heterogeneous as-received to a partially molten homogeneous structure with wustite particles in a slag phase was observed; from 1050 °C to 1150 °C for the acid and from 1050 °C to 1200 °C for the basic samples.

Therefore, it appears that the appearance of liquid in itself is not sufficient to cause softening. A sufficient amount of liquid that will promote the physical and chemical transformations in the pellets' microstructure from "asfired" to a semisolid is necessary. The liquid improves the mass transport of the heterogeneously distributed components. As temperature increases, so will the fraction of liquid, thus pushing the system further to equilibrium. After this amount has been formed, the system will tend toward equilibrium, regardless of the initial condition. Softening will occur after this transformation has begun, and other factors will also play a role. These, along with those findings presented in the two previous articles, will be discussed in the final article of this series.

ACKNOWLEDGMENTS

The authors acknowledge the Center for Iron and Steelmaking Research, Carnegie Mellon University (CISR), for the support for this project and Messrs. J. Black and P. Kaushik for help in preparing the samples. Paulo F. Nogueira also acknowledges the Institute for Technology Research of the State of São Paulo (IPT) for the support.

REFERENCES

- 1. P.F. Nogueira and R.J. Fruehan: *Metall. Trans. B*, 2004, vol. 35B, pp. 829-38.
- 2. P.F. Nogueira and R.J. Fruehan: *Metall. Mater. Trans. B*, 2005, vol. 36B, pp. 583-90.
- T. Bakker: Ph.D. Thesis, Delft University of Technology, Delft, The Netherlands, 1999.
- 4. P. Barnaba: Ironmaking and Steelmaking, 1985, vol. 12 (2), pp. 53-63.

- 5. J.V. Khaki, Y. Kashiwaya, and K. Ishii: *Ironmaking and Steelmaking*, 1980, vol. 21 (1), pp. 56-63.
- 6. V.J. Ritz, H.A. Kortmann, and K. Koch: *ICSTI/Ironmaking Conf. Proc.*, ISS, Warrendale, PA, 1998, pp. 1635-54.
- 7. L. Bentell: Scand. J. Metall., 1981, vol. 10, pp. 205-09.
- 8. T. Borider: Scand. J. Metall., 1987, vol. 16, pp. 122-28.
- 9. T. Borider: Scand. J. Metall., 1987, vol. 16, pp. 146-50.
- 10. S.M. Nutter: Ph.D. Thesis, Carnegie Mellon University, Pittsburgh, PA, 1989.
- C. Orrling, Y. Fang, N. Phinichka, S. Sridhar, and A.W. Cramb: JOM-e, 1999, vol. 51 (7), http://www.tms.org/pubs/journals/JOM/9907/Orrling/ Orrling-9907.html
- 12. H.A. Wriedt: *Binary Alloy Phase Diagrams*, 2nd ed., ASM International, Metals Park, OH, 1990, p. 1740.
- 13. P.F. Nogueira and R.J. Fruehan: Iron Steelmaker, 2003, vol. 7, pp. 29-35.
- 14. Slag Atlas, 2nd ed., Verlag StahlEisen, Düsseldorf, Germany, 1995, p. 79.