Induration Process of Pellets Prepared from Mixed Magnetite–35% Hematite Concentrates

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To explore the possibility of successfully processing iron ore pellets with a high content of hematite, thermogravimetric tests were performed to study the induration process of pellets composed of a mixture of iron ore concentrates (magnetite and 35% wt of hematite). Thermogravimetric tests were performed nonisothermally from 25°C to 1 400°C using two heating rates, 5 and 50°C/min. To identify the reactions involved and to follow the microstructural evolution throughout the induration process, selected tests were arrested at predetermined temperatures, and samples were rapidly cooled to room temperature for later characterization using X-ray diffraction (XRD) and scanning electron microscopy (SEM) along with energy dispersive spectroscopy (EDS). It was found that the formation of phases such as calcium ferrite (CF), magnesium ferrite (MF), silico-ferrites of calcium (SCF) and silico-ferrites of calcium and aluminum (SCFA) are influenced by the heating rate. The microstructure of the fired pellet processed at 50°C/min showed compact small grains of the sintered phase of the secondary hematite (SH), partially surrounded by a slag phase. In contrast, the fired pellet processed at 5°C/min exhibited a microstructure consisting of the SH phase with a faceted morphology surrounded by a relatively large amount of the slag phase. The results suggest that the pellet processed at 50°C/min had a more satisfactory response to the induration process.

KEY WORDS: iron ore; hematite; magnetite; pellet; pellet induration; sintering.

1. Introduction

In the process of making green pellets, binding agents such as bentonite are added to the iron ore concentrate to facilitate the formation of agglomerates. Additionally, additives such as dolomite and hydrated lime are also incorporated into the iron ore concentrate to promote suitable thermal processing of the green pellet “induration process”, which increases the mechanical resistance1-3) and can thus be used as a feed material in the blast furnace and in the direct reduction processes.3) The most well-known pellet forming practice consists of using concentrates with high magnetite content.1-4) The induration process is generally performed on a straight grate5) and is a complex operation because of the reaction phenomena occurring and the large number of process variables, such as the chemical composition of the pellet, the thermal profile, the partial oxygen pressure, and the height of the pellet bed, among others.

The induration process for green pellets involves 4 main stages: (1) drying, (2) preheating, (3) heating and (4) cooling. In the drying stage (105–260°C), water is removed from the pellet in a controlled process to avoid cracking. During preheating (260–845°C), oxidation of the magnetite and decomposition of carbonates occur.7) In the heating stage (845–1 329°C), the oxidation of the magnetite continues, and the formation of SCF begins along with the sintering of the particles of SH, which is formed by the oxidation of magnetite. It is accepted that the SCF formation is initiated by the solid-state reactions of CaO with SiO2 to form calcium silicate and of CaO with hematite to form CF.9-11) Then, the SCF forms from the reaction of CF and calcium silicate phases at approximately 1 000°C and in the presence of MgO promote the formation of MF. At 1 200°C, the remaining calcium silicate and the CF begin to melt, promoting the formation of SCF and this phase melts at approximately 1 250°C. The localized formation of SCFA12,13) is also possible in areas rich in Al2O3. SCFA is stable at approximately 1 300°C and will thus precipitate, and the remaining liquid phase will solidify in a vitreous phase of iron silicate and calcium silicate (slag phase). The sintering of SH particles occurs by grain growth and reduction of the surface area. The presence of a liquid phase helps the sintering process because it facilitates the migration of ions. At temperatures over 1 300°C, the hematite and SH are unstable and form magnetite,14) which adversely affects the sintered micro-
structure because the crystal structure transformation from hexagonal hematite to face-centered cubic magnetite.\(^{12,14–18}\)

Finally, in the cooling stage, the pellet is cooled to room temperature. The strength of the fired pellet depends on its microstructure, particularly on the characteristics of the sintered particle network and the distribution and quantity of the slag phase.\(^{19–23}\) The sintering of the particles of SH, which is a diffusional process, depends on the thermal profile of the induration process. While it is accepted that the presence of the liquid phase promotes the sintering process, the quantity of this phase must be controlled because it influences the characteristics of the slag phase found in the fired pellet. In turn, the amount of liquid phase depends on the chemical composition of the pellet, as determined by the ore, type and quantity of additives and the applied thermal profile. Currently, there is information available on the processing of pellets with high magnetite content and hematite content below 10%. It is well known that the hematite does not participate in the formation of the main binding phases SCF and SFCA. In addition, the hematite is less reactive than SH,\(^{14}\) and thus it does not intervene in the sintering process.

Nevertheless, iron ore with high magnetite content is becoming scarce, making its utilization more difficult. Therefore, there is an industrial interest in processing pellets made from mixtures of magnetite with significant amounts of hematite. In spite of the potential impact of this practice, there is very little research focused on the study of the induration process of this type of pellet, and the research that does exist has been conducted under very specific conditions.\(^{24,25}\) Therefore, the extrapolation to the industrial scale is difficult to assess. Successful processing of this type of pellet requires understanding the reactions that occur during the induration process and the evolution of the microstructure, which determines the strength of the pellet. In this context, this work studied the induration of pellets composed of a mixture of magnetite and 35% hematite using a thermogravimetric technique and emphasizes the reactions involved and the microstructural evolution.

## 2. Experimental Procedure

### 2.1. Materials and Elaboration of Green Pellets

The chemical compositions of the each of the materials used for this study are given in Table 1. In this paper, all compositions are given in weight percent. The contents of iron oxides in the iron ore were obtained using quantitative XRD. The XRD analysis showed minor contents of dolomite and calcium carbonate, 0.88 and 1.14% respectively. And the content of the others compounds was estimated from the elemental chemistry analysis performed using the Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) technique. While the chemical compositions of the binder (bentonite) and the additives (hydrated lime and dolomite) were obtained by qualitative XRD analysis and elemental chemical analysis by x-ray fluorescence.

Green pellets were produced in the laboratory using a dry mixture of iron ore (magnetite with 35% hematite), plus 4% dolomite, 1% bentonite and 1% hydrated lime. To the dry mixture, 10% water was added. The moist mixture was put into a rotating disc that was 20 cm in diameter to make the green pellets that were 1 cm in diameter. Prior to the induration process, the green pellets were dried at 110°C for 24 h in a muffle furnace.

### 2.2. Induration Testing

Figure 1 shows the experimental device used for the thermogravimetric testing of the pellets. The set up consists of a SETARAM Setsys Evolution16/18 thermobalance with 0.030 μg of resolution. The thermobalance has a computerized data acquisition system that records the temperature and the variation in mass during the induration process. For each induration test, the dry pellet was placed in a platinum wire basket and suspended from an arm of the balance. Then, the pellet was placed into the heating chamber. The temperature of the heating chamber was measured using a type S thermocouple (Pt-Pt/10% Rh) that was placed just underneath the hanging pellet. The tests were performed in...

![Fig. 1. Experimental set-up.](image)

### Table 1. Chemical composition of the raw materials.

<table>
<thead>
<tr>
<th>Code</th>
<th>Fe(_2)O(_3)</th>
<th>Fe(_3)O(_4)</th>
<th>SiO(_2)</th>
<th>FeO(OH)</th>
<th>TiO(_2)</th>
<th>Al(_2)O(_3)</th>
<th>SO(_3)</th>
<th>MgO</th>
<th>CaO</th>
<th>Na(_2)O</th>
<th>K(_2)O</th>
<th>LOI*</th>
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<td>Iron ore</td>
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<td>3.37</td>
<td>1.33</td>
<td>0.33</td>
<td>0.470</td>
<td>0.007</td>
<td>0.192</td>
<td>0.639</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Bentonite</td>
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<td></td>
<td>81.7</td>
<td></td>
<td>0.22</td>
<td>5.840</td>
<td>1.13</td>
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<td>2.18</td>
<td>2.11</td>
<td>0.73</td>
<td>0.282</td>
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<tr>
<td>Dolomite</td>
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<td></td>
<td></td>
<td>0.43</td>
<td>12.23</td>
<td>40.61</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>45.33</td>
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<tr>
<td>Hydrated lime</td>
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<td>0.09</td>
<td></td>
<td></td>
<td></td>
<td>0.03</td>
<td>0.32</td>
<td>69.22</td>
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<td>30.28</td>
</tr>
</tbody>
</table>

LOI*= Loss on ignition
an oxidizing atmosphere (dry air, 21% O₂-78% N₂) using a flow rate of 100 ml/min. First, the thermogravimetric tests were conducted at different heating rates (5, 30, 40 and 50°C/min) from 25°C to 1400°C. From the results obtained, the heating rates of 5 and 50°C/min were selected for additional tests, which were arrested at temperatures previously determined as a function of the variations in the trend of the evolution of mass observed in the previous tests. The processed pellets were microstructurally characterized and chemically analyzed. Additionally, tests were performed for each material used in the fabrication of the pellets to obtain information about their thermogravimetric behavior.

2.3. Derivative Thermal Analysis

The derivative thermal analysis method was used on the thermogravimetric curves obtained to more accurately determine the temperatures at which the pellets exhibit changes in the trend of the evolution of mass during the induration process. Figure 2 illustrates the thermogravimetric curve corresponding to the test conducted at 5°C/min, and the first and second derivatives are also included. In the same figure, dashed lines were placed at points where significant changes in mass occurred. The first derivative shows a minimum at 310°C, and then it increases and stays stable until 560°C, where it begins to decrease abruptly. A minimum at 775°C is also observed, which is followed by an increase and a progressive decrease to 1120°C. It is observed in Fig. 2 that the second derivative reaches a value of zero at the aforementioned temperatures. In view of these observations, tests were stopped at 310, 560, 775, 1120 and 1400°C. An additional test was carried out and stopped at 1200°C to obtain information about the evolution of the microstructure between 1120°C and 1400°C. The procedure was also applied to the tests conducted at 50°C/min with equivalent temperatures of 390, 600, 919, 1160, 1200 and 1400°C.

2.4. Microstructural Characterization and Chemical Composition

Pellets obtained from the arrested thermogravimetric tests were cut in half. One half was used for microstructural characterization and the other to determine quantities of magnetite and hematite. For microstructural characterization, the fired pellets were mounted in resin. Samples were put through the screening operation using 500–2400 grit silicon carbide paper with ethanol as a lubricant, and finally, they were polished with 3 and 1 μm diamond paste. To identify semi-quantitatively the phases present in each sample, SEM was used, PHILIPS XL30 ESEM, along with the EDS technique. The contents of magnetite, hematite and other compounds, such as SCF, MF and CMS, in the fired pellets were determined using the XRD in a PHILIPS X'Pert diffractometer with a copper anode source and analyzed with PCAPD version 4 h software. The preparation of the fired pellets consisted of grinding followed by screening using a 100 ASTM sieve. The identification of the crystalline phases was performed by comparing the XRD patterns with standards from the International Centre for Diffraction Data (ICDD). The Rietveld method was used in the FullProf program, which consists of the theoretical adjustment of the diffraction pattern by applying a model that includes structural factors and analysis conditions.

3. Results

Figures 3(a)–3(c) show the thermogravimetric curves obtained at the heating rate of 5°C/min for bentonite, hydrated lime hydroxide and dolomite. The dashed lines

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*Fig. 2.* Thermogravimetric curve with 1st and 2nd derivatives.

*Fig. 3.* Thermogravimetric curves for (a) bentonite, (b) hydrated lime and (c) dolomite at 5°C/min.
indicate significant variations in mass, and they were included to assist in later discussion on the reactions that occur during the induration pellet process.

Figure 4 shows thermogravimetric curves for pellets processed using heating rates of 5, 30, 40 and 50°C/min. The trend of the curves is qualitatively similar independent of the heating rate. However, as the heating rate increases, the curves shift to higher temperatures and lower values in changes in mass. Based on this behavior, the heating rates of 5 and 50°C/min were selected to study the chemical and microstructural evolution during the induration process of the pellets.

Figure 5 shows the variation of the %Fe3O4/%Fe2O3 ratio as a function of temperature for pellets processed at 5 and 50°C/min. It is thought that the onset of the magnetite oxidation reaction to produce hematite occurred below 310 and 390°C for 5 and 50°C/min, respectively, because of the slight decrease observed in the %Fe3O4/%Fe2O3 ratio at these temperatures. This result agrees well with the temperature range reported in the literature, 200–400°C.1,28–32 The oxidation of magnetite extends significantly as the temperature increases up to 775°C for 5°C/min and to 919°C for 50°C/min. The evolution of the magnetite oxidation reaction was confirmed by XRD of the pellets processed at 5°C/min and stop temperatures of 25, 310 and 775°C (Fig. 6). At 25°C, the major phases are magnetite and α-hematite. At 310°C, the intensity of the magnetite peaks decrease compared with those observed at 25°C, while peaks associated with the γ-hematite are identified. At 775°C, the peaks intensity corresponding to the magnetite are lower compared at 310°C; however, the intensity of the peaks corresponding to the α-hematite phase increased as a result of magnetite oxidation and the transformation of γ-hematite to α-hematite. In the range of 775–1200°C and 919–1200°C for 5 and 50°C/min, respectively, the %Fe3O4/%Fe2O3 ratio varies slightly. At 1200°C, the content of hematite for 5°C/min is 90% while it is 100% for 50°C/min (Table 2), contrary to what would be expected because at 5°C/min, the time available for the magnetite oxidation reaction is longer. This observation suggests that the phases formed, hematite and SCF, react with each other to form magnetite in the range of 1000–1200°C, which is promoted by the larger amount of SCF, as expected by the longer reaction time. It is important to note that a larger amount of the SCF phase is specified at 1120°C in Table 2 for the pellet processed at 5°C/min compared with the contents at 1160°C for the heating rate of 50°C/min. The analysis of the thermogravimetric curves to be presented later suggests that the magnetite phase forms at 1120 and 1160°C for the pellets processed at 5 and 50°C/min, respectively, which agrees relatively well with that reported in the literature, i.e., that the formation of magnetite occurs from 1200°C in the presence of Ca and Mg.28,31 In the 1200–1400°C range, the %Fe3O4/%Fe2O3 ratio increases for both heating rates, which is more evident in the pellet processed at 5°C/min and...
is assumed to be associated with the greater amount of liquid phase promoted by the lower heating rate; the liquid phase reacts with hematite to form magnetite. The presence of a larger quantity of liquid phase will be made evident in the later section concerning the microstructural analysis of the pellets.

The thermogravimetric curves shown in Fig. 7 exhibit a progressive decrease in mass from 25°C up to 290, 310 and 390°C in the case of the iron ore processed at 5°C/min and pellets processed at 5 and 50°C/min, respectively. This decrease in mass is associated with the removal of residual water and with goethite dehydroxylation, which starts at about 200°C.33–38 This result is confirmed by XRD analysis reported in Table 2, where the presence of goethite is indicated at 25°C while this phase is no longer detected at 310 and 390°C. In addition, the decrease in mass is greater in the pellets due to dehydration of bentonite, which is added during its fabrication, as shown in Fig. 3(a). In the temperature range of 290–614°C for the iron ore and 310–560°C and 390–600°C for the pellets processed at 5 and 50°C/min, respectively, the curves show an increase in mass due to the oxidation of magnetite, as shown in Fig. 5. The increase in mass is smaller in the pellets due to the mass loss generated by the dehydroxylation of bentonite (Fig. 3(b)) and calcium hydroxide (Fig. 3(b)) present in the pellets, which counters the mass gained by the oxidation of magnetite. The increase in temperature over the aforementioned ranges produces a decrease in mass in spite of the mass gain associated with the oxidation of magnetite. This decrease in mass is related to the decomposition of calcium carbonate and the descarbonatation of the dolomite present in the iron ore and the pellet. For the pellet the decrease in mass is principally to the descarbonatation of the dolomite which is added during its elaboration. The decomposition of calcium carbonate start approximately at 630°C,39,40 while the descarbonatation of dolomite occurs between 600 and 750°C, as indicated by the dashed lines in Fig. 3(c). The descarbonatation of dolomite is confirmed in Table 2 where it is observed that its content decreases as the temperature increases for both heating rates, resulting in an accentuated mass loss in the pellet, as observed in Fig. 7. As the decomposition of calcium carbonate and descarbonatation of dolomite progress, the mass loss becomes less important, and thus the curves present an increase in mass produced by the magnetite oxidation. From 1080°C for the iron ore and from 1120 and 1160°C for the pellets processed at 5 and 50°C/min, respectively, the mass decreases up to 1400°C, which is assumed to be associated with the reaction of hematite with the liquid phase formed at high temperatures to form magnetite. It is important to mention that Forsmo28 has reported that the temperature for the formation of magnetite begins at approximately 1200°C by the formation of magnesium ferrous in pellets made with olivine additions and processed at

<table>
<thead>
<tr>
<th>% wt (°C)</th>
<th>Fe₂O₃</th>
<th>Fe₃O₄</th>
<th>SiO₂</th>
<th>FeO(OH)</th>
<th>CaCO₃</th>
<th>CaMg(CO₃)₂</th>
<th>SCF*</th>
<th>CMS**</th>
</tr>
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<tbody>
<tr>
<td>25 5°C/min</td>
<td>28.24</td>
<td>58.40</td>
<td>2.95</td>
<td>1.28</td>
<td>5.7</td>
<td>3.42</td>
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<td>–</td>
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<tr>
<td>310 5°C/min</td>
<td>32.23</td>
<td>52.04</td>
<td>3.31</td>
<td>–</td>
<td>3.74</td>
<td>8.78</td>
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<tr>
<td>560 5°C/min</td>
<td>57.44</td>
<td>29.22</td>
<td>2.62</td>
<td>–</td>
<td>3.84</td>
<td>6.88</td>
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<td>775 5°C/min</td>
<td>85.1</td>
<td>5.27</td>
<td>5.27</td>
<td>–</td>
<td>1.87</td>
<td>2.49</td>
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<td>–</td>
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<tr>
<td>1120 5°C/min</td>
<td>89.37</td>
<td>2.26</td>
<td>1.38</td>
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<td>–</td>
<td>4.53</td>
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<td>1200 5°C/min</td>
<td>90.82</td>
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<tr>
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<td>25 50°C/min</td>
<td>28.24</td>
<td>58.40</td>
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<td>3.42</td>
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<td>600 50°C/min</td>
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<tr>
<td>919 50°C/min</td>
<td>77.59</td>
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<td>1160 50°C/min</td>
<td>86.1</td>
<td>1.8</td>
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<tr>
<td>1400 50°C/min</td>
<td>81.89</td>
<td>18.11</td>
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</table>

SCF* Silico ferrites of calcium Ca₃Fe₂(SiO₄)₃
CMS** Akermanite Ca₂Mg(Si₂O₇)
5°C/min.

Figure 8 shows that the microstructure of a green pellet exhibits a structure containing mainly particles of iron ore concentrate, which are assumed to be magnetite (M) and hematite (H). Both types of particles are hardly distinguished from one another under SEM and EDS. Particles of dolomite (D) and hydrated lime (HL) are also observed to a lesser extent.

Figure 9 shows the microstructure of pellets processed up to 775 and 919°C at 5 and 50°C/min, respectively. The heating rate does not promote significant changes in the microstructure because both microstructures are similar. The microstructure consists predominantly of particles with a rough surface that are assumed to be SH produced by the oxidation of the magnetite in agreement with Fig. 5. Additionally, a few particles with irregular shapes and smooth surfaces, similar to several found in Fig. 8, were observed. It is thought that these particles correspond to hematite and that it remains unalterable during the induration process. A silicon oxide phase (S) is also observed, which is assumed to be present in the iron ore.

The microstructures resulting from the heating to 1120 and 1160°C at 5 and 50°C/min, respectively, do not show significant differences from one another. Figure 10 shows the microstructure corresponding to the heating up to 1160°C at 50°C/min, which displays SH, H, S and MF particles inside of SH particles. Furthermore, Fig. 11 shows a magnification of a SH particle in which precipitates of MF are observed, which indicates that the formation of MF occurs in the temperature range of 775–1120°C and 990–1160°C at 5 and 50°C/min, respectively. It has been reported in the literature16,21,41–46) that MF is formed by the migration of Mg²⁺ toward the SH lattice in the range of 1000–1200°C and that depends on the atmosphere and the Fe³⁺/Fe²⁺ ratio.

Figure 12 corresponds to the microstructure of pellets
processed up to 1200°C. These microstructures show particles of SH, H, MF, S and SFCA. The presence of hematite particles indicates its stability at 1200°C, which is in agreement with that reported in the literature.14,47) The observed SCFA particles were formed below 1200°C between 1120 and 1160°C for 5°C/min and 50°C/min, respectively, which agrees well with that reported in the literature of approximately 1050°C.11) It is noteworthy that the SCFA phase is not detected by RXD at 1200°C, Table 2, because of the low quantity of this phase.

**Figure 13** shows the micrographs of pellets processed up to 1400°C. The resulting microstructure after processing the pellet at 5°C/min, Fig. 13(a), is characterized by a faceted SH phase surrounded by an abundant SCFA phase, whereas that corresponding to the pellet processed at 50°C/min, Fig. 13(b), exhibits an abundant SH phase characterized by small connected grains that are partially surrounded by the SCFA phase. As the heating rate increases, the driving force at high temperatures for sintering of the SH particle increases, thus promoting a massive growth and producing small grains connected to each other. In contrast, as the heating rate decreases, growth in a preferential direction is promoted, and a faceted morphology is formed. The amount of SCFA phase is a function of the time available to be formed, e.g., as the heating rate is increased, the time for formation of the SCFA phase is reduced, and thus this phase is present in smaller quantities. However, the presence of the SCFA phase promotes the formation of magnetite as it reacts with SH. Thus, the %Fe3O4/%Fe2O3 ratio at 1400°C, Fig. 5, is lower for the heating rate of 50°C/min.

4. **Conclusions**

(1) The microstructure of the pellet processed at 50°C/min suggests that pellets of magnetite and 35% hematite can satisfactorily react to the induration process. The microstructure was compact with small grains of sintered SH that were partially surrounded by a slag phase. In contrast, the resulting microstructure from the induration process at 5°C/min shows an SH phase with faceted morphology surrounded by a relatively large amount of the slag phase. At lower heating rates, the time for the diffusion process is favored, and larger grains were formed in comparison to those observed with a higher heating rate.

(2) The heating rate did not observably affect the microstructure of the pellet up to 1200°C. Significant changes were observed after the particle sintering process, particularly in terms of the size and morphology of the grains as well as the amount of slag phase formed.

(3) For the chemical composition of the pellet studied, the onset of the formation of magnetite at high temperature occurred at 1120 and 1160°C for heating rates of 5 and 50°C/min, respectively. The quantity of magnetite phase formed was greater for the process at 5°C/min because the time available for its formation was greater.
REFERENCES