Preliminary Evaluation of Pre-Reduction of Carbon-Based Titaniferous Magnetite Pellets for Reduction of Energy Requirements in the Vanadium and Steel Co-Production Process

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Smelting of titaniferous magnetite (titanomagnetite) for the co-production of steel and vanadium is an energy intensive process. A laboratory and desktop (Pyrosim) study was conducted to investigate pre-reduction of carbon-based titanomagnetite pellets for reduction of energy requirements in the overall process. Energy requirements for smelting untreated and pre-reduced titanomagnetite pellets at temperatures ranging between 900°C and 1200°C were determined. Degrees of Fe metallisation (DOM) and reduction (DOR) at respective temperatures were similar, and increased with increase in pre-reduction temperature, from below 20% at 900°C, to about 91% at 1100°C, and about 98% at 1200°C. Simulation results showed that pre-reduction at 900°C, 1100°C and 1200°C resulted in respective overall process energy savings of 8.2%, 4.8% and 3.8%. DOM and DOR of carbon-based titanomagnetite pellets at 900°C were low, thus a higher pre-reduction temperature of 1100°C associated with higher energy savings and appreciable DOM and DOR would be recommended. High DOM and DOR result in higher process throughput, and reduction of the possibility of slag foaming during smelting.

INTRODUCTION

Titanomagnetite is typically smelted in electric arc or blast furnaces (EAF or BF) for the co-production of vanadium and steel. During the development of EAF technology for titanomagnetite smelting, it was believed that the use of conventional blast furnace technology would be unfeasible, owing to possible formation of high-melting reduced titanium species that would require a higher coke rate and would descend into the cohesive zone as unmelted solids (Bleloch, 1949). However, some years later, blast furnace technology was successfully commissioned for the smelting of titanomagnetite in China by Panzhihua Iron and Steel Corporation (Pangang) and later by Chengde Iron and Steel (Chengde), and in Russia by Nizhny Tagil Iron and Steel (NTMK) (Goso, et al., 2016; Steinburg, 2011). Since the late 1960s, the now-defunct Evraz Highveld Steel and Vanadium Corporation (EHSV) in South Africa was one of the world-leading producers of vanadium and steel products from the smelting of the South African titanomagnetite containing relatively high vanadium content using EAF. Smelting of titanomagnetite for co-production of steel and vanadium is an energy intensive process (Steinburg, 2011). The demise of this major South African titanomagnetite smelter was partly due to unreliable supply and the high cost of electricity in South Africa. Reduction of energy consumption in the titanomagnetite smelting process for co-production of steel and vanadium has the potential to revive the titanomagnetite smelting industry in South Africa.

Mintek has developed an improved ilmenite smelting technology, known as Impril, which is essentially a pre-reduction and smelting process of carbon-based ilmenite pellets for the reduction of the overall energy consumption in the ilmenite process (Bisaka, et al., 2016). The current study adopted the pre-reduction principles used in the Impril technology for the processing of titanomagnetite.
The aim of the study was to investigate the feasibility of pre-reduction of carbon-based titanomagnetite pellets for the reduction of the overall energy consumption in the titanomagnetite smelting process.

**Overview of Impril technology**

Impril technology involves the inclusion of the pre-reduction of carbon-based ilmenite pellets before EAF smelting process, with a view of reducing the electric energy intensity of the overall process. In the Impril technology, ilmenite, carbon-based reductant, and organic binder are blended and subsequently agglomerated into pellets. The carbon-based ilmenite pellets are subjected to reduction at 900–1100°C, in the presence of CO or synthetic (CO+H₂) gas. Hot pre-reduced ilmenite pellets are then smelted in an EAF (Bisaka, et al., 2016).

The main challenges encountered during the smelting of ilmenite using either DC or AC furnace technologies to produce valuable titania slag and pig iron include high-energy requirements, production of rich CO gas that typically result in foaming of the slag, and aggressive behaviour of titania (TiO₂) slag against refractory lining of EAF. In particular, the foaming of titania slag during the smelting process poses a risk of blowing the roof off furnace building. Hence present ilmenite smelters operate with high roof tops. High roof tops lead to higher capital cost and higher energy losses. Pre-reduction of ilmenite operates on principles of removal of oxygen contained in the ilmenite component of the pre-reduction feed, through the addition of carbonaceous reductant, which reacts with oxygen to form CO gas. This minimises the amount of CO gas generated during the smelting process; thus reducing the risk of undesired slag foaming (Bisaka, et al., 2016).

Demonstration of Impril technology at Mintek indicated that high and consistent DOM and DOR of about 90%, high TiO₂ grades of more than 86%, and up to 40% overall ilmenite process energy savings can be achieved (Bisaka, et al., 2016).

**EXPERIMENTAL PROCEDURES**

**Materials and equipment**

Titanomagnetite ore mined from the main magnetite layer of South Africa’s Bushveld Complex was supplied by EHSV. The ore was subjected to elementary concentration by low-intensity magnetic separation at Mintek for another project. It is this concentrate that was used in the current study. The bulk chemical composition of the titanomagnetite concentrate is given in Table 1. Low ash petroleum Sasol carbon (sascarb) was used as reductant. Chemical grade CaO, MgO, Al₂O₃ and SiO₂ were used as synthetic fluxes. The chemical compositions of the synthetic fluxes and the reductant are also included in Table 1. A proprietary organic binder, ALCOTEC CB6 (CB6) binder was supplied by BASF.

<table>
<thead>
<tr>
<th></th>
<th>Titanomagnetite ore</th>
<th>Titanomagnetite concentrate</th>
<th>Sascarb</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe₃O₄*</td>
<td>77.7</td>
<td>77.9</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>MgO</td>
<td>1.48</td>
<td>1.34</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.97</td>
<td>4.17</td>
<td>98</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>1.34</td>
<td>1.25</td>
<td>98.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>0.07</td>
<td>&lt; 0.05</td>
<td>96</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TiO₂</td>
<td>13.3</td>
<td>13.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>V₂O₅</td>
<td>1.67</td>
<td>1.86</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>0.20</td>
<td>&lt; 0.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MnO</td>
<td>0.24</td>
<td>0.30</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>98.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Ash</td>
<td>0.33</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LOI**</td>
<td>0.97</td>
<td>0.5</td>
<td>3.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
*Total Fe expressed as Fe$_3$O$_4$; **loss on ignition; < 0.05 mass% indicates that concentration was below the limit of detection

Bulk chemical analyses of testwork samples were conducted at Mintek using Varian Vista-PRO CCD simultaneous inductively coupled plasma optical emission spectrometer (ICP-OES) and CS744 LECO combustion instrument.

Reduction roasting tests were conducted using a chamber furnace. The dimensions of the chamber furnace are 175 mm height, 195 mm width and 175 mm depth. The furnace chamber is equipped with six MoSi$_2$ elements that hang along the lining of the chamber which is made up of an alumina refractory board that is thermally stable up to 1800°C. Thus, the heating elements of the furnace are exposed to the sample inside the chamber. The reaction chamber is sealed using a solid-steel shell that is equipped with water-cooled rubber seals on the front door, and top opening for accessing elements and reaction gas inlet. The chamber of the furnace is gas-tight, thus the reaction atmosphere can be controlled. The furnace is also equipped with a gas-bubbler connected to the gas outlet of the chamber to observe that a positive pressure inside the reaction chamber is maintained during tests.

**Sample Preparation**

Reductant carbon addition was calculated using Equations [1] and [2]. Stoichiometric carbon addition was maintained at 110% as used in previous work (Maphutha, et al., 2017).

\[
\begin{align*}
\text{Fe}_3\text{O}_4 + 4\text{C} & \rightarrow 3\text{Fe} + 4\text{CO} & [1] \\
\text{V}_2\text{O}_5 + 5\text{C} & \rightarrow 2\text{V} + 5\text{CO} & [2]
\end{align*}
\]

For a relatively low temperature operation, a CaO:MgO ratio of 22:8 in the titaniferous slag was targeted. CaO, MgO, Al$_2$O$_3$ and SiO$_2$ were added to achieve the slag composition with liquidus temperature shown by the dashed-lines on the phase diagram in *Figure 1* (Jochens, 1967). This slag composition is the development slag composition proposed for the beneficiation of South African titanomagnetite through fluxing with quartz, limestone and magnesite (Bleloch, 1949).

Carbon-based titanomagnetite pellets were prepared by weighing and mixing the feed materials. Table 2 presents the mixing ratios used to produce the pellets. The binder type and addition used were adapted from the development work of Impril technology (Bisaka, et al., 2016). CB6 binder addition was maintained at 0.5% to the mass of titanomagnetite.
Figure 1. Equilibrium phase diagram for titaniferous slags in the compositional range: TiO$_2$ = 37.19%, SiO$_2$ = 19.69%, and Al$_2$O$_3$ = 13.12%, at varying ratios of CaO (30-0%) and MgO (0-30%) (adapted from Jochens, 1967).

Table 2. Normalised mixing recipe for preparation of carbon based titanomagnetite pellets (mass %)

<table>
<thead>
<tr>
<th>Titanomagnetite</th>
<th>Sascarb</th>
<th>CB6 Binder</th>
<th>CaO</th>
<th>SiO$_2$</th>
<th>MgO</th>
<th>Al$_2$O$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>74.0</td>
<td>1.7</td>
<td>0.4</td>
<td>6.0</td>
<td>4.5</td>
<td>1.1</td>
<td>0.4</td>
</tr>
</tbody>
</table>

**Pelletisation**
Carbon-based titanomagnetite micro-pellets with a size range of -5+2 mm were prepared using the mixture in Table 2. Pelletisation process involved forming small agglomerates in a rotating disc, with the growth of pellets aided by spraying water to the rolling material in the disc. The procedure entailed gradual addition of small portions of the recipe to the pelletising disc rotating at 16 rotations per minute while mounted on a tilted frame at an angle of 48°. The rotation of the disc causes the wetted fines to form small seed-like nuclei of particles which grow into pellets as they pick up small loose grain particles. The optimum point was reached when the pellets reached the desired size of -5+2 mm. Undersize and oversize pellets were hand crushed and recycled back to the disc.

**Physical strength testwork**
The strength tests were conducted on wet pellets and pellets dried at 105°C overnight in an oven. The respective batches of pellets were subjected to compressive and drop weight tests to assess their physical strengths. Compressive strength tests were conducted using a 10 kN Instron machine, which determined the maximum force required to fracture each pellet. The drop weight tests were conducted to determine the number of drops the respective pellets were able to withstand before breaking when dropped from a height of 1.0 m. A total of ten pellets were tested and the average values computed.
Pre-reduction
The aim of the pre-reduction tests was to determine DOMs and DORs at different temperatures. To complete each test, a 100 g mass of dry pellets was transferred into a high purity alumina crucible. The charged crucible was placed inside the chamber furnace. The chamber was sealed and purged with inert (Ar) gas for 10 minutes. The furnace was heated up at a rate of 10°C/minutes to the test temperature in Ar atmosphere. At temperature, the Ar flow was replaced with CO flow which remained for 60 min at the test temperature. At the end of test duration, the CO gas flow was replaced with Ar gas flow, furnace power turned off, and then the furnace allowed to cool down to room temperature. The reduction test temperatures were 900˚C, 1100˚C, and 1200˚C. Tests conducted at 900˚C were duplicated in order to evaluate the repeatability of the test procedure.

Subsamples of the respective batches of pre-reduced carbon-based titanomagnetite pellets were subjected to bulk chemical analyses using ICP-OES and LECO combustion for determination of carbon content, and iron speciation analyses for the determination of Fe\(^{3+}\), Fe\(^{2+}\) and metallic Fe using a method involving selective dissolution of target species and determination by ICP-OES.

Calculation of DOR and DOM
A mass balance of oxygen associated with each iron before and after pre-reduction was conducted. Oxygen associations with iron were determined using the oxidation states of the aforementioned metal. An assumption was made that only iron oxides would undergo reduction, i.e. only iron will lose oxygen during the reduction tests. Reduction of V and Ti oxides were assumed to be negligible under the solid-state reduction roasting conditions.

Oxygen balance was based on an assumption that the mass of oxygen associated with each gram of iron (Fe\(^{2+}\) or Fe\(^{3+}\)) in the untreated titanomagnetite is equal to the sum of the masses of oxygen associated with each gram of iron after reduction and oxygen removed during reduction of titanomagnetite. DOR was therefore calculated using Equation [3]

$$\text{DOR} = \frac{m_2}{m_1} \times 100$$

Where $m_1$ = total mass of oxygen associated with iron in the untreated titanomagnetite, i.e. total oxygen mass removable from titanomagnetite, $m_2$ = mass of oxygen removed during reduction.

Degree of metallisation is defined as the extent of conversion of Fe oxide into metallic Fe relative to total Fe in the processed carbon-based titanomagnetite pellets (Bleloch, 1949). Pre-reduced pellets were subjected to selective dissolution of metallic iron in a methanol-bromine lixiviant, followed by quantification of the dissolved Fe content using ICP-OES. The results obtained were used to determine the degree of iron metallisation calculated using Equation [4].

$$\%\text{DOM} = \frac{Fe^0}{Fe_{\text{total}}} \times 100$$

Where $Fe^0$ = metallic iron concentration, and $Fe_{\text{total}}$ = total iron concentration

PyroSim simulation
The Mintek process simulation software, PyroSim (Jones, 1987) was used to estimate the specific energy requirements (SER) for smelting of untreated titanomagnetite, and for pre-reduction (at previously specified temperatures) and smelting of carbon-based titanomagnetite pellets at 1600°C. Pre-reduced pellets were assumed to be hot-charged into the follow up smelting furnace. The total energy requirement for smelting of pre-reduced charge included the energy requirements for solid-state pre-reduction stage. A comparison of the results for the untreated and pre-reduced pellets was made.

For PyroSim calculations, feed rates of reductants and fluxes were based on 1000 kg/h of titanomagnetite concentrate. Heat losses of 0 kWh in pre-reduction and smelting units were assumed in all the calculations.
RESULTS AND DISCUSSION

Pelletisation
Physical compressive strengths of the carbon-based titanomagnetite pellets were checked against physical strengths specifications for iron ore pellets.

Compression Test Results
Wet and dried compressive strength specifications for iron ore pellets are 10 N and 22 N, respectively (Mbele, 2012). Average compressive strengths of wet and oven-dried carbon-based titanomagnetite pellets were 13 N and 22 N, respectively. Thus, wet and dry carbon-based titanomagnetite pellets would be suitable for transportation and handling under the same conditions as typical wet and dry iron pellets.

Drop Weight Test Results
Wet iron ore pellets typically withstand at least two drops from a height of 1.0 m before breaking. On average, wet titanomagnetite pellets withstood three drops from 1.0 m height before breaking. After drying, the pellets still withstood an average of three drops from 1.0 m height before breaking. The lack of improvement on drop numbers after drying was attributed to possible zero net strength gain as a consequence of an increase of strength by removal of free moisture and loss of strength due to thermal destabilisation of the CB6 organic binder.

Pre-reduction

Chemical composition of untreated and pre-reduced pellets
Chemical compositions of the untreated and pre-reduced carbon-based titanomagnetite pellets are given in Table 3. Results duplicate tests at 900°C were averaged to simplify reporting. The duplicate results were generally similar. Chemical analysis results showed that the metallic Fe (Fe⁰) content in the pre-reduced titanomagnetite pellets increased with pre-reduction temperature. At 900°C, Fe²⁺ content in the pellets increased significantly indicating that Fe³⁺ was reduced to Fe²⁺, while the metallic Fe content was still low. Increasing the reduction temperature resulted in significant decrease in the Fe³⁺ and Fe²⁺ concentrations, while the metallic Fe concentration increased significantly in the titanomagnetite pellets. These results suggested that the reduction of iron in the carbon-based titanomagnetite pellets moved from Fe³⁺ to Fe²⁺ and to Fe⁰. The iron reduction mechanism results observed in the current study agree with results reported in literature (She, et al., 2013).

Table 3. Chemical compositions of untreated and pre-reduced titanomagnetite pellets (mass %)

<table>
<thead>
<tr>
<th>Pre-reduction</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>Cr₂O₃</th>
<th>MgO</th>
<th>MnO</th>
<th>SiO₂</th>
<th>TiO₂</th>
<th>V₂O₅</th>
<th>Fe</th>
<th>C</th>
<th>Fe³⁺</th>
<th>Fe²⁺</th>
<th>Fe⁰</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>29.3</td>
<td>29.3</td>
<td>29.3</td>
<td>2.43</td>
<td>0.26</td>
<td>5.13</td>
<td>9.28</td>
<td>1.26</td>
<td>41.5</td>
<td>13.4</td>
<td>29.3</td>
<td>12.2</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>900°C</td>
<td>14.5</td>
<td>14.5</td>
<td>14.5</td>
<td>2.64</td>
<td>0.49</td>
<td>5.58</td>
<td>9.95</td>
<td>1.35</td>
<td>44.6</td>
<td>12.4</td>
<td>14.5</td>
<td>24.8</td>
<td>5.27</td>
</tr>
<tr>
<td>1100°C</td>
<td>0.15</td>
<td>0.15</td>
<td>0.15</td>
<td>3.02</td>
<td>0.27</td>
<td>6.74</td>
<td>12.6</td>
<td>1.64</td>
<td>55.4</td>
<td>4.86</td>
<td>0.15</td>
<td>4.67</td>
<td>50.6</td>
</tr>
<tr>
<td>1200°C</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>3.12</td>
<td>0.26</td>
<td>6.67</td>
<td>12.9</td>
<td>1.70</td>
<td>57.6</td>
<td>3.14</td>
<td>&lt;0.05</td>
<td>1.03</td>
<td>56.6</td>
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</table>

<0.05 mass% indicates that concentration was below the limit of detection

DOR and DOM
Figure 2 shows graphical results of the effects of pre-reduction temperature on DOM and DOR. The results show that DOM and DOR increased with pre-reduction temperature. The same phenomena was observed from the studies conducted at Mintek on the pre-reduction of ilmenite pellets (Bisaka, et al., 2016).

DORs achieved at 900°C, 1100°C and 1200°C were 17%, 91% and 98% respectively. DOM achieved at 900°C, 1100°C and 1200°C were 12%, 91% and 98% respectively.
The results showed that the pre-reduced carbon based titanomagnetite pellets had similar and excellent pre-reduction and metallisation degrees of about 91% and 98% when operating at temperatures of 1100°C and 1200°C, respectively. The fact that the DOM and DOR are similar at 1100°C and 1200°C, shows that the reduction of iron at these temperatures was mainly driven by and resulted primarily in the reduction of ferrous to metallic iron. In agreement with Bisaka et al., (2016), high DORs and DOMs at high temperatures were attributed to evaporation of the organic binder leaving behind pours on the pellets. The reducing CO gas diffused through these pours to effectively reduce and metallise the iron species around the respective micro-pellets (Bisaka, et al., 2016).

![Graphical results of DOR and DOM at different pre-reduction temperatures](image)

**Figure 2. Graphical results of DOR and DOM at different pre-reduction temperatures.**

**Pyrosim simulation**

Table 4 gives a summary of the results obtained from the Pyrosim simulations. The results show that SER for smelting of untreated carbon-based titanomagnetite pellets was 1170 kWh. The total energy required for pre-reduction and smelting of carbon-based titanomagnetite pellets were 1074 kWh, 1114 kWh and 1125 kWh at 900°C, 1100°C and 1200°C respectively. The results show that pre-reduction of carbon based titanomagnetite pellets at 900°C and smelting of the hot pre-reduced charge would result in process energy savings of 8.2%.

When the carbon-based titanomagnetite pellets with the same composition as the feed to the direct smelting approach were pre-reduced at 1100°C and subsequently hot fed in the smelting furnace, overall process energy savings of 4.8% were achieved. When the carbon-based titanomagnetite pellets were pre-reduced at 1200°C, the energy saving for the pre-reduction and smelting process were 3.8%.

In addition to the energy savings promoted by pre-reduction, the inclusion of a pre-reduction stage resulting in high DORs would offer benefits in terms of increased throughput at the smelting stage, and reduction of the possibility of the foaming phenomenon, which is normally experienced in operations involving titania-bearing slags.
Table 4. Results obtained from the Pyrosim simulations and the total percentage energy savings

<table>
<thead>
<tr>
<th></th>
<th>Untreated</th>
<th>900°C</th>
<th>1100°C</th>
<th>1200°C</th>
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<tbody>
<tr>
<td><strong>Mass of carbon-based</strong></td>
<td>1332</td>
<td>1332</td>
<td>1332</td>
<td>1332</td>
</tr>
<tr>
<td><strong>pellets, kg</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td><strong>Pre-red energy, kWh</strong></td>
<td>0</td>
<td>762</td>
<td>958</td>
<td>1010</td>
</tr>
<tr>
<td><strong>Mass of smelter feed</strong></td>
<td>1332</td>
<td>1040</td>
<td>972</td>
<td>960</td>
</tr>
<tr>
<td><strong>kg</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Smelting energy, kWh</strong></td>
<td>1170</td>
<td>312</td>
<td>156</td>
<td>115</td>
</tr>
<tr>
<td><strong>Total energy requirement for process, kWh</strong></td>
<td>1170</td>
<td>1074</td>
<td>1114</td>
<td>1125</td>
</tr>
<tr>
<td><strong>(pre-red + smelting energy)</strong></td>
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<td></td>
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<tr>
<td><strong>Energy savings,%</strong></td>
<td></td>
<td>8.20</td>
<td>4.80</td>
<td>3.80</td>
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</table>

CONCLUSION

Preliminary laboratory experiments and simulation studies were conducted to investigate pre-reduction of carbon-based titanomagnetite pellets for the reduction of energy in the overall co-production of vanadium and steel. During pre-reduction at temperatures ranging between 900°C and 1200°C (excluding 1000°C, in 100°C steps), similar DOMs and DORs were observed. DOMs and DORs increased with increasing pre-reduction temperature, from below 20% at 900°C to about 91% at 1100°C, and about 98% at 1200°C. High DOM and DOR at 1100°C and 1200°C showed that the reduction of iron at these temperatures was mainly driven by and resulted primarily in the reduction of ferrous to metallic iron. Pyrosim simulation results showed that pre-reduction at 900°C resulted in the highest overall process energy savings of 8.2%, while pre-reduction at 1100°C and 1200°C resulted in respective overall process energy savings of 4.8% and 3.8%. DOM and DOR of carbon-based titanomagnetite pellets at 900°C were low. For more beneficial operation, a higher pre-reduction temperature of 1100°C, which is associated with higher energy savings and appreciable DOM and DOR, is recommended. High DOR would result in high smelting process throughput and reduction of the possibility of slag foaming.

REFERENCES


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