Title: IMPROVED ILMENITE SMELTING PROCESS

Abstract: A method of preparing a pre-reduced ilmenite ore for smelting, wherein metal oxides contained in the ore are selectively reduced in solid-state reactions over titanium oxide, the method including the step of pre-reducing carbon-based pellets of the ore.


Published:
— with international search report (Art. 21(3))
— before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))
BACKGROUND OF THE INVENTION

[0001] This invention relates to a consolidated process for the preparation of carbon-based ilmenite pellets, the solid-state reduction thereof, and the subsequent smelting thereof in an electric furnace.

[0002] The smelting of ilmenite consumes substantial quantities of electrical energy. Additionally, the operability of the furnace can be hindered due to frothing effects.

[0003] Feed to the smelting furnace is generally made up of raw ilmenite ore and a solid, carbonaceous reductant. Raw ilmenite, in a particular process, is replaced by pre-reduced ilmenite pellets. The steps for the production process of the latter are to prepare ilmenite pellets using bentonite and to pre-reduce the pellets in a rotary kiln in the presence of a solid, carbonaceous reductant in excess. The smelting of the pre-reduced ilmenite pellets is thus carried out in an AC furnace. The TiO₂ slag produced in this way is, however, contaminated with the bentonite which is an inorganic binder.

[0004] An object of the present invention is to provide an alternative process for pre-reducing an ore essentially targeting the metallisation of iron oxides contained in the ore.

SUMMARY OF THE INVENTION

[0005] The invention provides a method of preparing a pre-reduced ilmenite ore for smelting, wherein metal oxides, such as iron, chromium and manganese oxides contained in the ore
are selectively reduced in solid-state reactions over titanium oxide, the method including the
step of pre-reducing carbon-based pellets of the ore.

[0006] The metal oxides, other than titanium oxides, in the pellets may be pre-reduced to a
maximum extent i.e. essentially fully or they may be partially pre-reduced.

[0007] The pellets may be less than 6mm in size and preferably lie in the range of 2mm to
5mm.

[0008] The pellets may be prepared from a blend of required proportions of the ore, coal
fines of -106 microns and a suitable organic binder.

[0009] The ratio of the coal to the metallic oxide content may be practically determined. For
example a stoichiometric ratio for the full reduction of iron in the ore can be used.

[0010] The organic binder content may lie in the range of 0 to 1%. This content may be
dictated by the physical properties of the resulting pellets principally the strength of the
pellets in a green state and in an air-dried or indurated state. The pellets may be air-
indurated for at least 4 days. This period is usually adequate to ensure that the pellets are
sufficiently strong to allow their safe and efficient handling to subsequent pre-reduction
reactors. The mechanical strength of the pellets is preferably above 600N. The pellets
should also have an acceptable behaviour in a hot reactor environment to avoid decrepitation
due to excessive swelling.

[0011] A single binder or a mixture of binders may be used. The invention is not limited in
this respect.
Pre-reduced pellets are evaluated based on the reduction extent of iron oxides contained in the ore. Preferably the iron oxide should be present in a quantity of less than 10% from the initial content. However a consistent pre-reduction yield should be a main target during a normal and stable operation.

The pellets may be subjected to a thermal reduction process or to a hybrid, solid-state, reduction process.

The pellets, air-dried and indurated, may be heated in a fixed bed reactor at an optimal residence time which may lie in a range of from 0.5 to 4 hours.

If a thermal pre-reduction step is adopted then the pellets may be heated at a temperature in the range of 1100 to 1200°C.

If the hybrid, solid-state, pre-reduction step is adopted then the pellets may be heated to a temperature in the range of 900 to 1000°C in a controlled atmosphere of a reducing gas.

The reducing gas may comprise one or more of the following: CO, syngas (CO + H₂), natural gas and hydrogen.

If a fixed bed reactor is employed then the reducing gas may be filtered through a hot burden in the reactor. The reducing gas flowrate should be selected to achieve an adequate reduction yield of the iron oxides in the ore, as well as acceptable reactor operation performance.
The invention finds particular application in the preparation of pre-reduced, carbon-based, ilmenite micro-pellets which are to be smelted e.g. in a DC open arc furnace. However, the principles of the invention may be employed for the pre-reduction of pellets of titaniferous magnetite, ferrochrome and ferromanganese ores for the subsequent production of titania slag, chrome and manganese, alloys respectively.

Reference has been made to heating the air-dried pellets in a fixed bed reactor. This is exemplary only and non-limiting. A moving bed and a rotary kiln may be employed in place of the fixed bed reactor, in a pre-reduction stage. It is important that abrasion of the pellets is minimised and it should be possible to separate pre-reduced fines from other material, for example through the use of magnetic or equivalent techniques.

BRIEF DESCRIPTION OF THE DRAWING

The invention is further described by way of example with reference to the accompanying figures wherein;

Figure 1 illustrates in flow chart form the pre-reduction of carbon-based, ilmenite micro-pellets and the subsequent smelting thereof;

Figure 2 is a diagram depicting an impact of the residence time on pre-reduction and metallisation degrees at 1000°C and 0.5 l CO/min; and

Figure 3 is a diagram depicting an impact of the CO flowrate on the pre-reduction and metallisation degrees at 1000°C and 1 h residence time.
DESCRIPTION OF PREFERRED EMBODIMENT

[0022] The invention is hereinafter described with reference to the pre-reduction of carbon-based, ilmenite, micro-pellets. Although this is a preferred application of the principles of the invention it is possible to adapt the principles described herein for the pre-reduction of titaniferous magnetite, ferrochrome and ferromanganese ores.

[0023] Raw ilmenite ore 10 of a suitable size is fed to a blender 12. The blender also receives coal fines 14 of -106 micron in size and an organic binder 16 formed from a single binder or from a mixed binder composition.

[0024] The ratio of the input coal to the ilmenite is determined taking into account practical considerations. For instance a stoichiometric ratio which achieves a full reduction of iron in the ilmenite ore can be used. Further, the input of organic binder or mixes of organic binders, in the range of up to 1%, is dictated by the physical properties of the resulting pellets, particularly the green and air-dried strengths of the pellets. The resulting pellets should also have an acceptable behaviour (subsequently) in a hot reactor environment to avoid decrepitation due to excessive swelling.

[0025] The blender 12 produces carbon-based, ilmenite, micro-pellets of 2mm to 5mm in size. These pellets are then air-dried (step 20).

[0026] The air-dried, indurated pellets are then subjected to a thermal pre-reduction step 22, or to a hybrid, solid-state pre-reduction step 24. In each instance the air-dried indurated
pellets are heated in a fixed bed reactor 26 for an optimal residence time, generally from 0.5 to 4 hours.

[0027] If use is made of the thermal pre-reduction process the pellets are heated in the reactor 26 to a temperature in the range of 1100 to 1200°C. If use is made of the hybrid approach then the pellets are heated in the reactor 26 to a temperature of 900 to 1000°C in a controlled atmosphere of a reducing gas 30 which comprises one or more of CO, syngas, natural gas and hydrogen. The reducing gas is filtered through the hot burden of the pellets in the reactor 26. The reducing gas flowrate is regulated to achieve an adequate pre-reduction yield. The flowrate should also be regulated to optimise the reactor operation, principally the thermal efficiency and the production cost.

[0028] Process parameters of importance, in respect of the of pre-reduction technique used, include: the ilmenite grain size distribution, the composition of the pellets, the sizes of the pellets, the operating temperature, the residence time and the reducing gas flowrate.

[0029] Taken under consistent operating conditions each method is able to produce a consistent pre-reduction yield. The hybrid method, despite operating at a lower temperature then the thermal reduction method, appears to offer a higher pre-reduction yield than the thermal method.

[0030] The fully or partially pre-reduced ilmenite pellets 32, emerging from the reactor 26, can be fed, cold or hot, to a conventional ilmenite smelting process 34.
Without being bound by the following explanation it is believed that the organic binder provides a more intimate contact between the ilmenite and the coal fines. The small pellet size feature, in a highly reducing atmosphere, assists the transfer of heat and mass in the diffusion of gaseous reductants, such as CO and H₂, to the reaction sites. The organic binder burns off at the process temperature, a feature which induces localised reduction and promotes the formation of cracks and pores in the ilmenite ore grains contained in the pellets. The specific surface areas of the ilmenite pellets are therefore increased and the diffusion rate of the gas reductant to the reaction sites is enhanced. This in turn impacts on the pre-reduction yield. The reduction process can be smoothly and efficiently operated despite the minor sintering of the pellets that may occur at elevated temperatures.

The fully or partially pre-reduced, carbon-based ilmenite pellets which are fed, either hot or cold, into a DC open arc furnace decrease the consumption of electricity in the furnace, help to address slag foaming and result in an improved grade of TiO₂ slag output by the furnace.

Through tests it has been established that iron oxide in the pellets was nearly completely reduced through the use of the hybrid pre-reduction process carried out at a temperature of 1000°C and for a residence time of 2 hours. The pre-reduction yield was increased as temperature, residence time and reducing gas flowrate were increased.

The use of the thermal pre-reduction process at a temperature of 1100 to 1200°C produced a pre-reduction yield of about 85% - a value which is adversely affected with an increase in ilmenite ore grain size and with an increase in the size of the coal fines.
About 4 tons of cold pre-reduced ilmenite pellets were smelted in a DC open arc furnace. The energy consumption of the furnace lay in the range of 0.6 to 0.7 kWh / kg of pre-reduced ilmenite pellets - a figure which represents an electrical energy saving of 30 to 40% compared to a conventional ilmenite smelting process. The smelting process was stable with no visible sign of foaming. The product 36 contained about 95% TiO\textsubscript{2} and about 3% FeO.

A higher grade TiO\textsubscript{2} slag (above 90%) can thus be achieved, using conventional ilmenite feedstock in smelting operations, with no foaming occurring. Using the method of the current invention, a lower grade ilmenite could be used as feedstock to produce TiO\textsubscript{2} slag of at least 85% TiO\textsubscript{2} content.

The invention has been described with reference to the use of a gaseous reductant. It is possible though to make use of a solid reductant such as anthracite or coal, instead of the reducing gas 30. Also the reactor 26 which, typically, is a fixed bed reactor can be replaced by a moving bed or by a rotary kiln configuration provided abrasion effects between the pellets are minimised. It should be possible though to separate the pre-reduced pellets, for example using magnetic techniques, from the other material emerging from the reactor.

Additional carbonaceous solid reductant can be used in excess to reduce residual iron in the slag to below 6% without inducing slag foaming.
DESCRIPTION OF A PILOT TEST OF THE PROCESS OF THE INVENTION

[0039] A 200 kW DC arc furnace facility was used for demonstrating the smelting of pre-reduced ilmenite pellets. The furnace had a 1 m outer diameter, water-spray cooled steel shell lined with a single layer and three rows of magnesite-chrome bricks and a hearth lined with rammable magnesia. The refractory lining resulted in the furnace crucible internal diameter (ID) of 0.656 m. The furnace was equipped with an alumina lined conical roof and a shell bolted on a domed base. A single taphole was used to tap a stream of both molten slag and metal. The furnace was equipped with a single and centrally-located graphite electrode of 40 mm diameter operating as a cathode while the anode comprised steel pins buried in the hearth. The feed system comprised individual hoppers used to feed anthracite and pre-reduced ilmenite pellets through a furnace feed pipe. The furnace was equipped with an off-gas system for the cleaning of produced process gas prior to release thereof into the atmosphere.

[0040] Carbon-based ilmenite pellets containing the as received ilmenite, stoichiometric amount of anthracite, were prepared using a proprietary organic binder at a required dosage. The as-received ilmenite had a particle size distribution of $D_{10}$ in the 38 µm to 150 µm size range. The anthracite was milled to a $D_{SS}$ passing 106 pm to facilitate its incorporation into an ilmenite pellet recipe. Pellets were prepared in a pilot-scale pelletizing unit comprising an inclined rotating pan of 985 mm diameter and 170 mm depth. The mechanical strengths of the pellets were measured and found to vary with the type and dosage of binder used, within a range of 0.01 - 0.03 MPa for green pellets and 0.81 - 1.50 MPa for indurated pellets at ambient conditions.
[0041] Batches of 250 kg each of indurated pellets were reduced in an electrically heated muffle furnace operated at a controlled temperature of 1100 °C. During a three hour firing time, in total 5 kg of CO was blown intermittently through the reactor burden at intervals of 10 minutes. The pellets were loaded in a single tray of 1700 mm x 900 mm, having a loading area of a mesh screen acting as a distribution plenum for the reducing gas.

[0042] Both the raw and pre-reduced ilmenite materials at various conditions were chemically analysed; specifically an analysis of the iron oxidation states (Fe$^{3+}$, Fe$^{2+}$, and Fe$^0$) was used to calculate the pre-reduction and metallisation yields for the pellets. Negligible reduction of titanium oxides was assumed throughout and the pre-reduction yield was therefore calculated based on the mass balance of oxygen associated with each gram of iron before and after pre-reduction. Equations [1] and [2] were used for the calculation of the prereduction and metallization yields, respectively.

\[
\text{Pre-reduction yield } = 100 \times \frac{\text{Oxygen removed by the pre-reduction process}}{\text{Oxygen associated with each gram of iron in the ilmenite sample}} \quad [1]
\]

\[
\text{Metallisation yield } = 100 \times \frac{\text{Fe}^0}{\text{Fe}_{\text{metal}}} \quad [2]
\]

[0043] The chemical analyses of the ore and anthracite are summarised in Table 1 and Table 2, respectively.
Table 1: Bulk chemical composition of the raw ilmenite (mass %)

<table>
<thead>
<tr>
<th></th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>CaO</th>
<th>TiO₂</th>
<th>V₂O₅</th>
<th>Cr₂O₃</th>
<th>MnO</th>
<th>FeO</th>
<th>Fe²⁺</th>
<th>Fe⁰</th>
<th>Fe/Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.28</td>
<td>0.44</td>
<td>0.57</td>
<td>0.05</td>
<td>46.6</td>
<td>0.25</td>
<td>0.09</td>
<td>1.08</td>
<td>47.87</td>
<td>13.50</td>
<td>&lt;0.05</td>
<td>1.33</td>
</tr>
</tbody>
</table>

<0.05%: the analyte concentration could not be accurately quantified as it is below the limit of detection (LOD).

Total Fe in the sample is expressed as % FeO.

Table 2: Summary of the bulk chemical composition of the anthracite (mass %)

<table>
<thead>
<tr>
<th>Moisture</th>
<th>Ash</th>
<th>Volatile</th>
<th>Fixed carbon</th>
<th>Total carbon</th>
<th>Total sulphur</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.57</td>
<td>4.74</td>
<td>6.19</td>
<td>89.1</td>
<td>90.7</td>
<td>0.56</td>
</tr>
</tbody>
</table>

[0044] In total, about 3.6 tons of pre-reduced pellets were produced. The pellets were bagged in 1 m³ bags from which five composite samples were collected. The chemical analyses of the 5 composite samples are given in Table 3.

Table 3: Chemical compositions of the pre-reduced pellets

<table>
<thead>
<tr>
<th></th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>CaO</th>
<th>TiO₂</th>
<th>V₂O₅</th>
<th>Cr₂O₃</th>
<th>MnO</th>
<th>Total Fe</th>
<th>Fe⁰</th>
<th>Fe²⁺</th>
<th>C</th>
<th>Ti³⁺</th>
<th>Fe/Ti ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>TP Bag 1</td>
<td>0.53</td>
<td>0.33</td>
<td>0.31</td>
<td>0.10</td>
<td>44.4</td>
<td>0.36</td>
<td>0.07</td>
<td>1.05</td>
<td>34.92</td>
<td>25.55</td>
<td>9.37</td>
<td>7.23</td>
<td>6.55</td>
<td>1.32</td>
</tr>
<tr>
<td>TP Bag 2</td>
<td>0.55</td>
<td>0.30</td>
<td>0.26</td>
<td>0.07</td>
<td>44.5</td>
<td>0.36</td>
<td>0.08</td>
<td>1.06</td>
<td>35.23</td>
<td>25.44</td>
<td>9.79</td>
<td>7.72</td>
<td>6.76</td>
<td>1.32</td>
</tr>
<tr>
<td>TP Bag 3</td>
<td>0.50</td>
<td>0.30</td>
<td>0.24</td>
<td>0.15</td>
<td>43.5</td>
<td>0.33</td>
<td>0.07</td>
<td>1.03</td>
<td>33.60</td>
<td>21.90</td>
<td>11.7</td>
<td>7.97</td>
<td>5.45</td>
<td>1.29</td>
</tr>
<tr>
<td>TP Bag 4</td>
<td>0.48</td>
<td>0.32</td>
<td>0.39</td>
<td>0.11</td>
<td>42.4</td>
<td>0.33</td>
<td>0.07</td>
<td>1.05</td>
<td>34.46</td>
<td>22.66</td>
<td>11.8</td>
<td>8.01</td>
<td>4.78</td>
<td>1.35</td>
</tr>
<tr>
<td>TP Bag 5</td>
<td>0.36</td>
<td>0.45</td>
<td>0.64</td>
<td>0.18</td>
<td>41.9</td>
<td>0.33</td>
<td>0.07</td>
<td>1.08</td>
<td>36.01</td>
<td>24.21</td>
<td>11.8</td>
<td>7.20</td>
<td>4.66</td>
<td>1.43</td>
</tr>
<tr>
<td>Average</td>
<td>0.48</td>
<td>0.34</td>
<td>0.37</td>
<td>0.12</td>
<td>43.3</td>
<td>0.34</td>
<td>0.07</td>
<td>1.05</td>
<td>34.84</td>
<td>23.95</td>
<td>10.89</td>
<td>7.63</td>
<td>5.64</td>
<td>1.32</td>
</tr>
<tr>
<td>St dev</td>
<td>0.07</td>
<td>0.06</td>
<td>0.16</td>
<td>0.04</td>
<td>1.17</td>
<td>0.02</td>
<td>0.004</td>
<td>0.02</td>
<td>0.90</td>
<td>1.64</td>
<td>1.21</td>
<td>0.39</td>
<td>0.98</td>
<td>0.02</td>
</tr>
</tbody>
</table>
The calculated degrees of prereduction and metallization for the five composite samples are presented in Table 4.

![Table 4: Pre-reduction and metallisation degrees of ilmenite pellets](image)

Tables 3 and 4 show that pellets prereduced to a consistent extent were produced as a result of the uniform furnace operating conditions.

Results from laboratory tests in a tube reactor of 80 mm diameter showed a very important feature of this process that is presented in Figures 2 and 3. Tests conducted at a temperature of 1000°C, showed that pre-reduction and metallisation degrees are both related to the residence time and CO flowrate. Increasing the CO flowrate appears to positively affect the yields, suggesting that CO diffusion would play a significant role in this process.
Continuous smelting of partially reduced ilmenite pellets (approx. 70% prereduction yield) was carried out to demonstrate stable furnace operation as well as production of a consistent slag quality, in particular, a slag TiO₂ grade above 85%. The test work also had the objective of confirming the process specific energy requirement. The slag results are presented in Table 5.

<table>
<thead>
<tr>
<th>Tap</th>
<th>TiO₂</th>
<th>Cr₂O₃</th>
<th>MnO</th>
<th>FeO</th>
<th>V₂O₅</th>
<th>SiO₂</th>
<th>CaO</th>
<th>Al₂O₃</th>
<th>MgO</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>84.74</td>
<td>1.34</td>
<td>1.37</td>
<td>2.06</td>
<td>0.30</td>
<td>1.31</td>
<td>0.34</td>
<td>1.96</td>
<td>6.58</td>
</tr>
<tr>
<td>B</td>
<td>87.84</td>
<td>0.60</td>
<td>1.18</td>
<td>2.59</td>
<td>0.21</td>
<td>1.16</td>
<td>0.28</td>
<td>2.49</td>
<td>3.65</td>
</tr>
<tr>
<td>C</td>
<td>83.15</td>
<td>0.31</td>
<td>1.68</td>
<td>2.73</td>
<td>0.29</td>
<td>1.20</td>
<td>0.38</td>
<td>2.76</td>
<td>7.50</td>
</tr>
<tr>
<td>D</td>
<td>86.66</td>
<td>0.45</td>
<td>0.89</td>
<td>4.89</td>
<td>0.17</td>
<td>0.49</td>
<td>0.31</td>
<td>1.45</td>
<td>2.70</td>
</tr>
<tr>
<td>E</td>
<td>88.19</td>
<td>0.45</td>
<td>0.99</td>
<td>4.37</td>
<td>0.16</td>
<td>0.43</td>
<td>0.36</td>
<td>1.53</td>
<td>3.53</td>
</tr>
<tr>
<td>F</td>
<td>91.27</td>
<td>0.31</td>
<td>1.47</td>
<td>1.58</td>
<td>0.15</td>
<td>0.28</td>
<td>0.32</td>
<td>1.47</td>
<td>3.16</td>
</tr>
<tr>
<td>G</td>
<td>94.28</td>
<td>0.08</td>
<td>1.32</td>
<td>1.25</td>
<td>0.10</td>
<td>0.21</td>
<td>0.17</td>
<td>0.98</td>
<td>1.61</td>
</tr>
<tr>
<td>H</td>
<td>93.32</td>
<td>0.09</td>
<td>1.21</td>
<td>1.48</td>
<td>0.10</td>
<td>0.16</td>
<td>0.14</td>
<td>1.10</td>
<td>2.40</td>
</tr>
</tbody>
</table>

*by difference

Table 6: Evolution of composition of pig iron from the stable smelting operation, in mass %

<table>
<thead>
<tr>
<th>Tap</th>
<th>Fe</th>
<th>Ti</th>
<th>V</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>C</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>95.54</td>
<td>0.67</td>
<td>0.24</td>
<td>0.34</td>
<td>1.04</td>
<td>0.48</td>
<td>1.66</td>
<td>0.02</td>
<td>0.01</td>
</tr>
<tr>
<td>B</td>
<td>95.92</td>
<td>0.43</td>
<td>0.23</td>
<td>0.36</td>
<td>1.08</td>
<td>0.44</td>
<td>1.53</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>C</td>
<td>95.15</td>
<td>0.55</td>
<td>0.16</td>
<td>0.38</td>
<td>1.47</td>
<td>0.33</td>
<td>1.92</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>D</td>
<td>97.03</td>
<td>0.16</td>
<td>0.09</td>
<td>0.18</td>
<td>0.52</td>
<td>0.21</td>
<td>1.76</td>
<td>0.02</td>
<td>0.03</td>
</tr>
<tr>
<td>E</td>
<td>94.59</td>
<td>0.29</td>
<td>0.20</td>
<td>0.62</td>
<td>1.14</td>
<td>0.51</td>
<td>2.57</td>
<td>0.03</td>
<td>0.05</td>
</tr>
<tr>
<td>F</td>
<td>91.81</td>
<td>3.64</td>
<td>0.23</td>
<td>0.64</td>
<td>1.17</td>
<td>0.86</td>
<td>1.62</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td>G</td>
<td>95.83</td>
<td>0.57</td>
<td>0.20</td>
<td>0.41</td>
<td>0.71</td>
<td>0.60</td>
<td>1.64</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>H</td>
<td>93.66</td>
<td>1.34</td>
<td>0.32</td>
<td>1.28</td>
<td>0.89</td>
<td>0.88</td>
<td>1.56</td>
<td>0.02</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Slag FeO contents as low as 1.3% were achieved without visible signs of slag foaming. This condition was maintained for a longer period during which stable furnace operation was demonstrated and slags of consistent FeO content were produced. Results for
this particular test work suggest that smelting of partially reduced ilmenite and operating the furnace with lower FeO content in the slag are technically possible.

[0050] The 200 kW DC open-arc furnace was operated at a power level in the range of 115 - 140 kW and at a corresponding voltage of 100 - 115 V. Consistent furnace heat losses in the range of 60 - 90 kW were measured. Average tapping temperatures measured using an optical pyrometer were scattered within a range between 1670 and 1780°C. The specific energy requirement (SER) for the smelting of prereduced carbon-based pellets was measured between 0.6 and 0.7 kWh / kg prereduced ilmenite. A 30-40 % reduction in furnace electricity required relative to a conventional smelting process can be achieved assuming that a prereduction yield of at least 70 % can be achieved. Arc resistivities were measured for various conditions investigated in order to predict the furnace arc stability. Arc resistivity was found to be in the range of 0.0168 and 0.0240 Q.cm which range is close to 0.0175 Q.cm, a typical value for arc resistivity in smelting processes with CO-rich atmospheres (in the absence of foaming).
CLAIMS

1. A method of preparing a pre-reduced ilmenite ore for smelting wherein metal oxides, contained in the ore are selectively reduced in solid-state reactions over titanium oxide, the method including the step of pre-reducing carbon-based pellets of the ore.

2. A method according to claim 1 wherein the metal oxides, other than titanium oxides, in the pellets are pre-reduced to a maximum extent.

3. A method according to claim 1 or 2 wherein the pellets are less than 6mm in size.

4. A method according to claim 3 wherein the pellets are between 2mm and 5mm in size.

5. A method according to any one of claims 1 to 4 wherein the pellets are prepared from a blend of the ore, coal fines and an organic binder.

6. A method according to claim 5 wherein the coal fines have a size of -106 microns.

7. A method according to claim 5 or 6 wherein the ratio of the coal to the metallic oxide content is determined using a stoichiometric ratio for the full reduction of iron in the ore.

8. A method according to any one of claims 5 to 7 wherein the organic binder content is in the range of 0 to 1%.

9. A method according to any one of claims 5 to 8 wherein a single organic binder or a mixture of organic binders is used.

10. A method according to any one of claims 1 to 9 wherein the pellets are air indurated for at least 4 days.
11. A method according to claim 10 wherein a mechanical strength of the pellets is of the order of 600N.

12. A method according to any one of claims 1 to 11 wherein iron oxide in the ore is present in a quantity of less than 10%.

13. A method according to any one of claims 1 to 11 wherein the pellets are subjected to a thermal reduction process or to a hybrid, solid-state, reduction process.

14. A method according to claim 13 wherein the pellets are heated in a fixed bed reactor for a time period of between 0.5 to 4 hours.

15. A method according to claim 14 wherein a thermal pre-reduction step is adopted and the pellets are heated to a temperature in the range of 1100 to 1200°C.

16. A method according to claim 13 or 14 wherein a hybrid, solid-state, pre-reduction step is adopted and the pellets are heated to a temperature in the range of 900 to 1000°C.

17. A method according to claim 16 wherein a reducing gas is filtered through a hot burden in the fixed bed reactor.

18. A method according to claim 17 wherein the reducing gas comprises one or more of the following: carbon monoxide; syngas (CO+H2); natural gas; and hydrogen.

19. A method according to any one of claims 1 to 18 wherein the smelting is conducted in a DC open arc furnace at a temperature of between 1650 and 1750°C.
Effect of CO flowrate on carbon-based pellets pre-reduction (1 hr, 1000°C)

FIGURE 2
Effect of time on carbon-based pellets pre-reduction (100% CO, 0.5 l/min, 1000°C)

Degree of reduction
Degree of metallization
Linear (Degree of reduction)

FIGURE 3
A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B1/245 C22B34/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>the whole document</td>
<td></td>
</tr>
<tr>
<td>X</td>
<td>CN 103 451 454 A (UNIV JIANGSU)</td>
<td>1-19</td>
</tr>
<tr>
<td></td>
<td>18 December 2013 (2013-12-18)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>the whole document</td>
<td></td>
</tr>
<tr>
<td>X</td>
<td>&amp; CN 1034 514 54K KI</td>
<td>1-19</td>
</tr>
<tr>
<td></td>
<td>18 December 2013 (2013-12-18)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>the whole document</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>US 3 765 868 A (MOKLEBUST 0 ET AL)</td>
<td>1-19</td>
</tr>
<tr>
<td></td>
<td>16 October 1973 (1973-10-16)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>the whole document</td>
<td></td>
</tr>
</tbody>
</table>

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents:

**A** document defining the general state of the art which is not considered to be of particular relevance

**E** earlier application or patent but published on or after the international filing date

**L** document which may throw doubts on priority claim(s) one of which is cited to establish the publication date of another citation or other special reason (as specified)

**O** document referring to an oral disclosure, use, exhibition or other means

**P** document published prior to the international filing date but later than the priority date claimed

**T** later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

**X** document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

**Y** document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

**Z** document member of the same patent family

Date of the actual completion of the international search: 24 April 2017

Date of mailing of the international search report: 08/05/2017

Name and mailing address of the ISA/
European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040
Fax: (+31-70) 340-3016

Authorized officer: Swiatek, Ryszard

Form PCT/ISA/210 (second sheet) (April 2005)
<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Patent document cited in search report</td>
<td>Publication date</td>
<td>Patent family member(s)</td>
</tr>
<tr>
<td>---------------------------------------</td>
<td>-----------------</td>
<td>-------------------------</td>
</tr>
<tr>
<td>US 6685761 B1</td>
<td>03-02-2004</td>
<td>NONE</td>
</tr>
<tr>
<td>CN 103451454 A</td>
<td>18-12-2013</td>
<td>NONE</td>
</tr>
<tr>
<td>US 3765868 A</td>
<td>16-10-1973</td>
<td>NO 130827 B</td>
</tr>
<tr>
<td>US 3765868 A</td>
<td></td>
<td>US 3765868 A</td>
</tr>
</tbody>
</table>