Pelletization and Direct Reduction of Local Iron Oxide

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Introduction:
This research project involved using a local iron ore from the Black Hills and reducing it to form iron and later steel for use in the making of a Japanese Samurai sword. In an attempt to gain a better grasp on the kinetics of reducing iron, a series of tests have been conducted to decide on a starting material and how long this selected material will need to reduce under different atmospheres and pellet composition.

Characterization of Ores:

Procedure
- Three different iron oxide concentrates were analyzed: Pacer, Homestake, and Cleveland Cliffs pellets
- Pacer had four different unit operations: Bulk, Joe Crusher, Shaker Table, Magnetic Separator
- Each Pacer sample was analyzed for mica content by x-ray diffraction (XRD) with particle size analysis
- The Homestake iron ore concentrate was a wet sludge dried and then it was analyzed with XRD
- A muffle furnace was used to run a reduction test on each ore. A sample of each was prepared and mixed with metallic coke and then placed in the blast furnace for 4 hours. Figure 1 shows an photograph of the three samples in the muffle furnace.

Results
- The Pacer sample focused on the reduction was the sample that had been magnetically separated. Although the sample had a very good particle size distribution, the mica content was still 38.3%
- The Pacer sample from the muffle furnace showed poor reduction and XRD showed most of the material had reduced to wustite and had about 7% iron.
- Initial XRD analysis showed that the Homestake iron oxide concentrate consisted of limonite. The Homestake sample from the muffle furnace had a good reduction and the XRD showed that the sample was basically 100% iron.
- The Cleveland Cliffs pellets were used only in the muffle furnace tests and had a very slow reduction. Only the outside edge of the pellets reduced to pure iron.

Agglomeration:
- Pelletizing experiments were conducted only on the Homestake ore based on the results of the reduction tests in the muffle furnace
- A 10 gallon Nalgene bottle was used as a tumbler
- Variables altered involved the way in which moisture was added, using bentonite, polyvinyl alcohol, and flour and sugar as binders, and varying the amount of binder added
- A meat grinder was tested as a means of making pellets
- Variables altered involved using dried material versus not dried material and varying the amount of binder for binding
- After determining the not dried material worked best, the water content of the sludge was determined as shown in Table 3
- Different pellet compositions and their relative strengths are shown in Table 1

Discussion of Results:
Pacer sample did not show much decrease in mica content from the bulk material to the material after it had been magnetically separated. The first muffle furnace test revealed that the Homestake iron oxide concentrate had the best percent reduction, therefore the Homestake sample was selected for pelletizing. When pelletizing in the 10 gallon Nalgene drum, it was hard to control pellet size because of the inability to evenly rehydrate the material. Pellets produced from the Nalgene shatter as are the pellets from the meat grinder only broke in half. This is possible because of the drying of the material. The initial particle size in the un-dried material allowed for the pellets to sinter better. It was determined that excess bentonite will hinder the reduction rate of iron oxide. Pellets sintered with 12% bentonite showed no reduction in 30 minutes of sintering. Pellets sintered with 3% bentonite exhibited some reduction in 30 minutes. Some thought had to go into why the final muffle furnace test did not melt the pellets. It is believed that not enough carbon was added to the crucibles in order to fully reduce the iron and not enough to get the iron to a 4.3% carbon content, which would lower the melting point to 1147°C melting easily in the muffle furnace.

Direct Reduction:

Procedure
- Experiments conducted in quartz tube, Figure 2, and muffle, Figure 1, furnaces attempting to reduce iron oxide pellets to iron.
- Variables altered in quartz tube tests were pellet composition and length of time in furnace. The factors for each test can be viewed in Table 1.
- Carbon monoxide and argon were used to create a reducing or inert atmosphere. Argon was used only with carbon infused pellets.
- All tests in quartz tube were conducted at 1000°C
- Experiment conducted in the muffle furnace involved using different types of pellets, one with and one without carbon.
- Reduction time was six hours at 1050°C and then temperature was increased to about 1200°C for an hour to achieve melting

Results
- In both the quartz tube and muffle furnace experiments, the carbon pellets fell apart at high temperatures.
- The 3% bentonite pellets held together well and exhibited the best reduction.
- The crucible that had carbon and pellets layered in alternating layers showed much better reduction than the crucible with carbon and pellets mixed together.
- The crucible with the layered carbon also yielded some metallic iron.

Discussion of Results:

Table 1. Pellet Composition, Sintering Process, and Pellet Strength

<table>
<thead>
<tr>
<th>Trial</th>
<th>Non Carbon, No Bentonite Pellets</th>
<th>No Carbon, 12% Bentonite Pellets</th>
<th>No Carbon, 3% Bentonite Pellets</th>
<th>Carbon Added Pellets</th>
<th>Furnace</th>
<th>Mass Wet %</th>
<th>Mass Ore %</th>
<th>Reduction Rate</th>
<th>Carbon Mortar</th>
<th>Reduction Gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>88.7</td>
<td>73.2</td>
<td>68.1</td>
<td>3.7</td>
<td>Forge</td>
<td>100 1000</td>
<td>70 70</td>
<td>60%</td>
<td>90 90</td>
<td>100 100</td>
</tr>
<tr>
<td>2</td>
<td>97.8</td>
<td>87.3</td>
<td>77.1</td>
<td>3.7</td>
<td>Forge</td>
<td>100 1000</td>
<td>70 70</td>
<td>60%</td>
<td>90 90</td>
<td>100 100</td>
</tr>
<tr>
<td>3</td>
<td>97.8</td>
<td>87.3</td>
<td>77.1</td>
<td>3.7</td>
<td>Furnace</td>
<td>100 1000</td>
<td>70 70</td>
<td>60%</td>
<td>90 90</td>
<td>100 100</td>
</tr>
</tbody>
</table>

Table 2. Iron Ore Sludge Moisture Content

<table>
<thead>
<tr>
<th>Initial Weight of Sludge (g)</th>
<th>Weight of dried soil (g)</th>
<th>Percent Water</th>
<th>Percent Ore</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Trial</td>
<td>402</td>
<td>104</td>
<td>74.1</td>
</tr>
<tr>
<td>2 Trial</td>
<td>258</td>
<td>64.2</td>
<td>75.7</td>
</tr>
<tr>
<td>3 Trial</td>
<td>270</td>
<td>62.7</td>
<td>77.3</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td>75.1</td>
</tr>
</tbody>
</table>

Table 3. Mass Reduction Experiments in Quartz Tube Furnace

<table>
<thead>
<tr>
<th>Trial</th>
<th>Description of Pellet</th>
<th>Time in °F</th>
<th>Gas Used</th>
<th>% Mass Lost</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Non Carbon, 12% Bentonite Pellets</td>
<td>750°F</td>
<td>Carbon Monoxide</td>
<td>12.15%</td>
</tr>
<tr>
<td>2</td>
<td>No Carbon, 12% Bentonite Pellets</td>
<td>1100°F</td>
<td>Carbon Monoxide</td>
<td>10.10%</td>
</tr>
<tr>
<td>3</td>
<td>No Carbon, 3% Bentonite Pellets</td>
<td>750°F</td>
<td>Carbon Monoxide</td>
<td>14.85%</td>
</tr>
<tr>
<td>4</td>
<td>No Carbon, 3% Bentonite Pellets</td>
<td>1100°F</td>
<td>Argon</td>
<td>26.74%</td>
</tr>
<tr>
<td>5</td>
<td>No Carbon, 3% Bentonite Pellets</td>
<td>750°F</td>
<td>Carbon Monoxide</td>
<td>20.56%</td>
</tr>
</tbody>
</table>

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