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 test 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 analyzed: Pacer, Homestake, Cleveland Cliffs pellets
- Pacer had four different unit operations: Bulk, Jaw 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 metallurgical 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 for 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 38.2%
- 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, poly vinyl 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 bentonite for binding
- After determining the not dried material worked best, the water content of the sludge was determined as shown in Table 2
- Different pellet compositions tested and their relative strengths are shown in Table 1

Figure 1. Image of crucibles in muffle furnace during first reduction test.

Figure 2. Image of quartz tube furnace setup.

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

Pellet Composition						Sintering Process			Description of Strength of the Pellet
Trial Number	Pellet Description	Mass Wet Sludge (g)	Mass Ore (g)	Bentonite Added (g)	Carbon Added (g)	Furnace or Forge	Temperature (°C)	Time (minutes)	
1	No Carbon, No Bentonite Pellets	88.7	21.6	0	0	Forge	900-1100	30	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.
2	No Carbon, 3% Bentonite Pellets	397.7	97.8	2.9	0	Forge	900-1100	30	After sintering looked as if they had reduced somewhat. These pellets were very strong. They could be droped from head height and would not break apart.
3	Carbon, 3% Bentonite Pellets	427.6	105.2	3.2	26.1	Furnace	700	75	These carbon pellets were extremely weak. When pressed on a bit, they crumbled apart into very small pieces.
4	No Carbon, 3% Bentonite Pellets	500.9	123.2	3.7	0	Furnace	750	75	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.
5	No Carbon, 3% Bentonite Pellets	1099	270.3	8.1	0	Furnace	750	90	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.

Table 2. Iron Ore Sludge Moisture Content

	Initial Weight of Sludge (g)	Weight of dried ore (g)	Percent Water	Percent Ore
Trial 1	402	104	74.1	25.9
Trial 2	258	64.2	75.2	24.8
Trial 3	270	62.2	77.1	22.9
		Average	75.5	24.5

Table 3. Mass Reduction Experiments in Quartz Tube Furnace

Trial #	Description of Pellets	Time in Furnace	Gas Used	% Mass Lost
1	Non Carbon, About 32% bentonite pellets	3hrs 20min	Carbon Monoxide	12.15%
2	Non Carbon, About 32% bentonite pellets	2hrs	Carbon Monoxide	10.38%
3	Non Carbon, No Bentonite Pellets	4hrs	Carbon Monoxide	14.85%
4	Carbon, 3% Bentonite Pellets	6hrs	Argon	26.74%
5	Non Carbon, 3% Bentonite Pellets	6hrs	Carbon Monoxide	20.56%

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 parameters for each test can be viewed in Table 3.
- 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 two 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.
- XRD analysis was conducted on some reduced particles from the layered crucible, but they produced mixed results and were inconclusive.

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 were as 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 determine that excess bentonite will hinder the reduction rate of iron oxide. Pellets sintered with 32% 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.

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