A LABORATORY STUDY RELATING TO THE PRODUCTION AND PROPERTIES OF PIG IRON NUGGETS

B. Anameric and S.K. Kawatra
Michigan Technological Univ.
Houghton, MI

ABSTRACT

Pig iron nuggets were produced in a laboratory scale furnace at Michigan Technological University. These nuggets are produced from pellets consisting of a mixture of iron oxide, coal, flux, and a binder, which are heated to 1450°C. These pellets then self-reduce to produce a solid, high-density, highly metallized (96.5% Fe) pig iron. During the nugget production process, a separate slag phase forms that clearly separates from the metal. The physical and chemical properties of the pig iron nuggets are similar to pig iron produced by blast furnaces, which is distinct from Direct Reduced Iron (DRI).

INTRODUCTION

Pig iron is predominantly produced by smelting iron ore in blast furnaces. The raw materials used in the blast furnace are pellets, flux, binder, and coke. Coke is added to the system as a fuel source, and is used because of its combination of reducing power and mechanical strength. The pellets for the blast furnace consist of iron ore concentrate that has been formed into balls, and the balls are sintered at approximately 1200° to harden them while converting the iron oxides to Fe2O3. Flux (limestone) to the blast furnace to promote strength. The pellets for the blast furnace consist of iron ore concentrate that has been formed into balls, and the balls are sintered at approximately 1200° to harden them while converting the iron oxides to Fe2O3. Flux (limestone) to the blast furnace to promote formation of a fusible liquid slag to collect impurities which will be separated from the metal.

There are a number of features of blast furnace operation which lead to inefficiency, increase costs, or lead to environmental problems:
1. When pellets are produced for blast furnace feed, they are first heated, then cooled for easy transportation and handling, and then reheated in the blast furnace for reduction and melting. Large amounts of energy are wasted in the process of cooling and re-heating.
2. Blast furnace operations are constrained by the need to use metallurgical grade coke. Due to the increasing regulations on environmental controls for coke production, and depletion of reserves of metallurgical coking coal, suppliers are becoming short and costs are increasing. Therefore as the cost of coke increases the blast furnace process becomes more costly.
3. Full thermal and chemical efficiency for a blast furnace process can only be achieved in large scale operations. This makes it difficult to rapidly react to incremental change in production capacity and market demands (Zervas et al, 1996).

The need for energy-efficient, smaller, economic, flexible, and environmentally friendly process units makes blast furnace operations unattractive, and encourages the development of new processes for production of iron from ore.

To overcome the disadvantages of the blast furnace process, a new one-step Iron Nugget Process was developed. Since coke is not used as a fuel for this process, it does not have the environmental impact of producing noxious chemical by-products of the coking process. In addition, energy is conserved due to reduction, carburization, melting, and slag separation occurring in one step. As a result, no energy is being lost due to cooling and reheating of the iron oxide feedstock.

This iron nugget process is capable of directly producing solid, high-density, highly metallized pig iron nuggets from dried green balls. These green balls are made out of fine iron bearing material, flux, binder, and reductant. The reductant (coal) is added to the system to supply the carbon required for reduction and carburization. Binder (Bentonite) in conjunction with the finely-ground ore particles serves to improve the properties of green balls in wet or dried condition. The flux is limestone, which fluidizes the slag and also prevents excessive iron losses in the slag.

The objective of this work was to: (1) verify the technical feasibility of the production of pig iron nuggets utilizing this unique process, (2) determine the important chemical and physical properties of these pig iron nuggets, and (3) determine the %Fe lost in the slag. During the experimental work the dried green balls were fired at furnace temperature 1450°C and residence time of 22 min.

EXPERIMENTAL

Raw Materials

Magnetite concentrate used in balling was obtained from an iron ore concentrator located in the Lake Superior district of the United States. The concentrate had a 10% moisture content, a particle size of 80% passing 25 microns (500 mesh), and contained 4.9% silicate gangue. The bentonite was Na montmorillonite based clay mined from the Western United States, by the American Colloid Company, and designated SPV 200. It had a plate water absorbance (PWA) of 706 (ASTM E946, 1996) and a particle size of 85% passing 74 microns (200 mesh) (Kawatra and Ripke, 2001). The chemical composition of the bentonite was: 34.62%SiO2, 23.16% Al2O3, 5.49% Fe2O3, 9.63% CaO, 2.11% MgO, 1.06% Na2O, 0.39% K2O, 1.25% TiO2, 2.20% P2O5, and 5.93%SO3. Limestone was used as a flux. Pulverized coal was used as a reductant. The coal sample was received from Electrical Power Research Institute (EPRI), and had the composition: 7.34% ash, 2.11% S, 30.73% volatiles, 72.89% C, 4.78% H, 1.30% N, and 5.11% O. Particle size analyses were performed by laser diffraction using a Microtrac SRA unit.

Balling Procedure

The balling procedure was developed to closely represent the
conditions that exist in full-scale plants. The balling feed mixture was prepared from magnetite concentrate with addition of 0.66% dry wt. bentonite clay as a binder, 7.5% dry wt. limestone as a flux, and 20% dry wt. coal. A Kneader-mixer (Readco Type-A mixer, 350-rpm with a 150-rpm orbital motion) was used to mix the feed mixture for five minutes. The feed mixture was delumped through a 2.4 mm (8 mesh) screen before balling. A laboratory scale-balling drum (overall diameter 40.6 cm (16 in), mouth diameter 25.4 cm (10 in) and depth 17.8 cm (7 in)) rotating in a counter-clockwise direction at 35 rpm was used for balling. Moistening a small amount of material with distilled water mist made seeds. After seeding, additional material and distilled water mist were added to make green balls. The green balls were removed from the drum periodically, and screened between 12.7 mm and 11.2 mm (1/2 x 7/16 in) in diameter. Finished greenballs were dried at 105°C (220 F) in Blue M (Illinois, USA) forced-air drying oven for 20 to 24 hours (Kawatra and Ripke, 2001). The apparent density of the dried green balls was 2.5 g/cm³.

Firing of Dried Greenballs

Dried green balls were fired in a Thermolyne 46100-type high temperature laboratory box furnace. Approximately 3.7 g of green balls were placed in graphite crucibles (4 cm diameter, 5 cm tall), which were transferred into the box furnace at 1450°C. After 22 minutes, the crucible was removed from the furnace and rapidly air-cooled to produce pig iron nuggets.

Analyzing the Pig Iron Nuggets

OPTICAL MICROSCOPY

Optical microscopy was used as a preliminary method to characterize the nugget microstructure and compare it with pig iron. Nuggets were mounted in epoxy resin, sectioned and polished (ASTM E3, 2001). Polished samples were etched in order to optically enhance microstructural features such as the grain size and other microstructural features. Nital (2%, nitric acid in methanol) was used as an etchant (ASTM E407, 1999).

APPARENT DENSITY AND POROSITY

Apparent density and density measurements were used to evaluate the degree of metallization. Apparent density measurements were made using the Archimedes principle (ASTM B311, 2002). Four nuggets were weighed twice, before and after being submerged in distilled water. The apparent densities of nuggets were calculated using Equation 1:

\[ \rho = \frac{M}{M - m} \]  
Eq. 1

Where \( \rho \) is the apparent density, \( M \) is the weight of the nugget, and \( m \) is the weight of the nugget submerged in distilled water. For these calculations the density of distilled water at room temperature is assumed to be 1.00 g/cm³.

The volume and the percent porosity of the nuggets were obtained using Cone Beam X-ray Microtomography (Lin and Miller, 2002). The density of the nuggets was correlated using the apparent density measurements and percent porosity. The correlation was made by using Eq. 2.

\[ D = \frac{\rho}{P} \]  
Eq. 2

Where \( D \) is the density, \( \rho \) is the apparent density, and \( P \) is the % porosity of the nugget.

Analyzing the Slag

The slag which separated from the pig iron nugget was ground using a tungsten carbide puck mill. The magnetic and non-magnetic materials in the ground slag were separated using a Davis Magnetic Tube separator. The magnetic materials separated were called magnetic slag and non-magnetic materials were called non-magnetic slag. The iron content of the ground slag, magnetic slag, and non-magnetic slag were determined by titration with potassium dichromate solution.

RESULTS AND DISCUSSION

Production of Nuggets

Pig iron nuggets were produced at 1450°C furnace temperature and 22 min residence times. The pig iron nuggets accompanied by their slag are shown in Figure 1. The slag was cleanly separated from the high density, highly metallized nuggets.

When the crucible was pulled out of the furnace, molten slag and molten nuggets were observed (see Figure 2.). The immiscibility and the density difference between the molten slag and the metal enabled the slag separation.

Microstructure of Pig Iron Nuggets

The microstructures of a pig-iron nugget is shown in Fig. 3 (a-c). It exhibits a structure characteristic of low C and Si white cast iron. (Davis, 1990) (Mehl, 1972) (Mampaey, 2001) (Heine and Barton, 1977) (Radzikowska and Voort, 1998). In the microstructure, the
darkest tree-like or fern-like areas represent former austenite dendrites (now pearlite) and light areas represent eutectic mixture. Unlike gray iron, malleable iron, and ductile iron, the microstructure seen here contains no free graphite. The presence of dendrites (tree-like or fern-like structures) indicates that melting of the metal did occur.

Apparent Density and Porosity Measurements

The average apparent density of four nuggets was 6.55 g/cm³ with a standard deviation of 0.51. The high standard deviation of the measurements was caused by variation in the amount of pores from one nugget to another. For the nugget shown in Fig. 4 the apparent density was measured to be 7.00 gr/cm³. For the same nugget, the % porosity was measured to be 3.31%. The density of the nugget calculated by correlated apparent density and porosity was 7.19 gr/cm³. This density value is comparable to the pig iron density of 7.2 gr/cm³ which is reported in literature (Weiss, 1985).

Chemical Analysis

The nuggets were analyzed by fusing into a bead of appropriate geometry, and then determining the elemental composition by X-ray fluorescence spectroscopy. The results are given in Table 1, and it is clearly seen that the nugget was very highly metallized, with over 96% iron.

Table I: Composition of the iron nuggets, as determined by X-ray fluorescence spectroscopy.

<table>
<thead>
<tr>
<th>Compound</th>
<th>%</th>
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<tr>
<td>Total Fe</td>
<td>96.49</td>
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<tr>
<td>Ni</td>
<td>0.01</td>
</tr>
<tr>
<td>MnO</td>
<td>0.12</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.03</td>
</tr>
<tr>
<td>CaO</td>
<td>0.92</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.05</td>
</tr>
<tr>
<td>S</td>
<td>0.96</td>
</tr>
<tr>
<td>SiO₂</td>
<td>0.88</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.86</td>
</tr>
<tr>
<td>MgO</td>
<td>0.52</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.12</td>
</tr>
<tr>
<td>As</td>
<td>0.01</td>
</tr>
<tr>
<td>Zn</td>
<td>0.00</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>0.03</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>38</td>
</tr>
</tbody>
</table>
Analyzing the Slag

The results of the experiments which were conducted for analyzing the slag are summarized in Table II. The slag contained 17.66% iron, of which 30% was magnetically recoverable. The remaining iron in the slag was in a non-magnetic form that could not be easily recovered.

Table II. summary of the results of magnetic separation and chemical analysis of the slag.

<table>
<thead>
<tr>
<th>Material</th>
<th>%Fe</th>
<th>Weight %</th>
<th>% Fe Recovery</th>
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<tbody>
<tr>
<td>Slag</td>
<td>17.66</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>Magnetic Slag</td>
<td>95.5</td>
<td>5.57</td>
<td>30</td>
</tr>
<tr>
<td>Non-magnetic Slag</td>
<td>13.1</td>
<td>94.43</td>
<td>70</td>
</tr>
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</table>

CONCLUSIONS

The laboratory experiments show that high density, highly metalized solid pig iron nuggets can be produced at a furnace temperature of 1450°C after a residence time of 22 minutes. Examination of the nugget microstructure showed that the nuggets were essentially identical to pig iron produced by conventional blast furnace processing. When the internal porosity of the nuggets was accounted for, the metal density was found to be 7.2 g/cm³, which indicates that the nuggets were almost entirely iron. This is supported by the chemical analysis of the nuggets, which determined that they were 96.49% total Fe. It was also determined that limestone flux additions resulted in a satisfactory slag that cleanly separates from the nuggets, removing silica and other impurities.

ACKNOWLEDGEMENTS

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REFERENCES

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