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Production of Sponge Iron from Pyrites

Robert W. Leeson

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PRODUCTION OF SPONGE IRON FROM PYRITES

By

Robert W. Leeson

A Thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

Montana School of Mines
Butte, Montana
May 1951
# PRODUCTION OF SPONGE IRON FROM PYRITES

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PRODUCTION OF SPONGE IRON
FROM PYRITES

INTRODUCTION

Since the livelihood of the world today is so dependent upon iron and steel, and since iron ore reserves are rapidly reaching depletion, the possibility of the production of metallic iron from pyrites should be studied. The purpose of this thesis is, therefore, to record and discuss the results of tests conducted, at the Montana School of Mines, in which pyrite concentrates from the Concentrator Department of the Anaconda Reduction Works, Anaconda, Montana, were subjected to the reducing effects of natural gas at elevated temperatures in an endeavor to produce iron in the metallic form.

The U. S. Bureau of Mines has conducted many tests over a period of the last 25 years in an attempt to devise a commercial process for the production of sponge iron from low-grade iron ore deposits which abound in this country but are not amenable to blast-furnace operations because of the excessive costs of production. It was from their numerous Reports of Investigations that the fundamental bases for this thesis work were obtained.¹

The results of the tests conducted proved very favorable with respect to the production of the base metal from pyrites. The methods and equipment employed were relatively crude but the results obtained indicated that further work should be carried on in order to study the possibility of founding a commercial process for sponge iron production.
from pyrites. Under existing economic conditions, it is highly doubtful that a process could be recommended which would allow for the operation of a plant, operating solely for the production of sponge iron from pyrites, however, it is possible that a feasible process could be developed in certain plants, where the pyrites are separated and roasted for their sulfur content, or where they are sent directly to waste. If this type of by-product operation could be effected, it would serve two purposes—it would serve in the recovery of strategic metal and also serve as a source of revenue from a heretofore discarded or waste product.

Because of the equipment which was used, the results of the tests should not be considered as exact facts but rather as good indications of what can be expected. The primary purpose of the research work was to determine if sponge iron could be obtained by the previously described method, and the apparatus used was a little crude for precise quantitative work. Each step in the procedure was quite time consuming because a uniformity of conditions among tests was desirable and required considerable supervision on the part of the operator.

EQUIPMENT

Standard laboratory equipment, such as, beakers, burettes, flasks, liter bottles, and reagents, were used in conjunction with an electric laboratory furnace and sillaminite tube which are illustrated in Fig. 1. The pyrites were roasted and charged into the furnace
and rabbled with a small steel rod or by a gentle, manual rotation of the tube. The gas was admitted from a standard gas outlet through a one-holed, rubber stopper, and the flow was regulated by the height of the flame from the exhaust end of the tube.

The furnace operated on 110 volts with a 200-ohm resistor in the circuit, so that, a fairly accurate temperature control could be maintained throughout the tests. The actual temperature of the furnace was obtained by means of a pyrometer of the thermocouple type, which was periodically inserted into the open end of the tube.

A small, gas-fired muffle furnace was used to dead-roast the pyrites prior to their admission to the reduction step.

**PROCEDURE**

In the first sets of tests, samples of dead-roasted pyrites were charged directly into the sillimanite tube and the charge was brought up to the desired temperature. The charge was rabbled to insure complete elimination of the sulfur content. If sulfur was still present in the ore, visible sulfur dioxide fumes could be detected as they escaped from the end of the tube. When it was apparent that the sulfur was eliminated, the natural gas was connected to one end of the furnace, and the excess was ignited as it left the other end. The gas was ignited for the reason of safety and also to insure a uniformity of flow for each test. In most cases, the gas burned at the mouth of the tube without its having to be ignited by the operator.
The charge was rabbled about every ten minutes because the gas would channel or flow directly over the top of the charge, and, since the tube was in a horizontal position, the possibility of gas penetration to the bottom of the charge was highly improbable if the rabling procedure was not followed.

Four variables were kept constant during the tests and a fifth varied each time. The size of the material, the rate of gas flow, the time of reduction, and the weight of sample, were kept constant, and the temperature was varied. In this way, the results of the tests could be used on a comparison basis.

The final products from the preliminary tests were tested rapidly and quite conclusively through the use of a magnet and also a copper-sulfate solution. These tests were strictly of a qualitative nature and at best only an indication of the product obtained.

A magnetic test was conducted on the dead-roasted product and then on the final product and from this test it was obvious that some metallic iron existed in the processed product because the final product showed a much greater affinity for the magnetic force.

For the copper-sulfate test, a sample of the final product was placed in a solution of copper sulfate and the copper deposit in the flask was considered conclusive proof that metallic iron existed in the product and that the process of reduction had been a relative success.

The next step in the procedure was to devise a method whereby quantitative tests could be conducted to determine the metallic iron-total iron ratio of the end product. The total
iron content was determined by the potassium-dichromate method. All of the iron was placed in solution with hydrochloric acid; the ferric iron reduced to ferrous iron with stannous chloride; mercuric chloride and a titrating solution of sulfuric acid, phosphoric acid, and water was added, with six to eight drops of sodium diphenylamine sulfonate as an indicator; and this solution was titrated with standardized potassium-dichromate solution. The dichromate solution was standardized with chemically-pure, metallic iron.

Upon the recommendation of the U. S. Bureau of Mines, the mercuric-chloride method for the determination of metallic iron was used to determine the metallic-iron content of the final product. In a dry, tightly-stoppered flask, a 0.2500-g, -65 mesh, sample of the reduced product was thoroughly mixed with 3 g of mercuric chloride salt. Since this procedure recommends that the flask be filled with carbon monoxide and none was readily available, the flask was filled with natural gas while the two constituents were being mixed. After the mixing step was finished, 50 milliliters of boiling water were added and swirled for one minute, and since the samples were badly carburized, the solution was boiled gently for one minute. At the completion of this phase, another 100 milliliters of boiling water were added, and the bottle was stoppered and placed in a lukewarm bath for about two minutes and then into an ice bath for rapid cooling. When the solution was cooled, the contents were filtered through a vacuum filter, the residue on the paper was washed with cold water and the clear, supernatant liquid was titrated immediately with 20 milliliters of the titrating solution, 6 - 8 drops of sodium di-
phenylamine sulphonate indicator, and the standard potassium dichromate solution.

For the standardization of the potassium-dichromate solution, three samples were processed, and in all the other tests, duplicate samples were run.

Extreme care was exercised in an endeavor to keep the reduced product in a reducing atmosphere. At the end of the reduction time, the sillimanite tube was removed from the furnace and the gas was allowed to flow through the tube until the charge was relatively cool. The reduced product was then placed in a stoppered flask which was also filled with natural gas, and in this manner, oxidation of the iron was greatly retarded. The mercuric-chloride method of analysis is quite sensitive, and since the samples had to stand about for some time between tests, these precautions were imperative if worthwhile results were to be obtained.

In the third type of tests which were performed, the pyrites were not dead roasted, but rather the sulfur content was reduced to about 6 per cent. The charge was placed in the sillimanite tube, and the tube was inserted into the furnace which was at about 800 F to prevent the breakage of the tube. The reducing gas was admitted immediately, and the rheostat resistance cut to nil so that the furnace temperature would rise as fast as possible. In this way, any oxidation, by the air, of the sulfur in the charge was minimized. As the temperature of the furnace rose, the charge was rabbled until the charge sintered. The sintering took place between 1500 and 1600 F. The sintered charge was then left dormant in the furnace for the
duration of the test. The last $\frac{1}{2}$ hour of the test, the furnace temperature reached 1800 F.

A different method of cooling the product was practiced in this test. Instead of passing gas through the tube as the charge cooled, the tube was removed from the furnace and the gas shut off. The gas stopper was left intact so that air could not readily circulate through the tube and oxidize the iron. By this cooling method, the final product was obtained in a light-gray color while the product obtained with the other cooling method was very heavily carburized.

The final product was assayed in the same manner as previously described for the second set of tests, and the sulfur content of the roasted product was determined by the barium-chloride precipitation method.$^2$

**OBSTACLES**

Several obstacles were encountered during the laboratory tests, however, in no instance did the obstacles seem insurmountable if revisions in the equipment used, were to be made.

As previously stated in this paper, the equipment which was used (Fig. 1) was rather crude and was used to determine if a reduction could be effected, rather than, a device to conduct precise quantitative tests. Since it was proved that the process worked, more adaptable apparatus could be designed and used.

The reduced products which were obtained were badly carburized, and in the cases of the longer periods of reduction, the carburization
was much greater than with the samples of the shorter reduction periods. Because of these highly-carburized samples, dissolution of the samples was quite difficult, even when aqua regia was used and the samples were or had to be subjected to many hours of slow digestion on a hot plate. In order to compensate for this high degree of carburization, the mercuric-chloride determinations for the metallic-iron content of the sponge-iron samples were boiled gently for an additional minute prior to the ice-bath treatment. This additional boiling procedure was practiced with only the two-hour reduction products.

The temperature control of the furnace proved rather spasmodic, and it was also found that when the gas was admitted to the tube, the temperature inside the tube dropped approximately 100 degrees below the temperature before the gas line was attached. The type of furnace and temperature control used did not allow for rapid adjustments in temperature, and the temperature fluctuation upwards and downwards was of 25 degrees. This condition proved almost unavoidable since it was impossible to leave the pyrometer inside the furnace for the entire duration of the tests because the charge could not be rabbled manually with the pyrometer in place. If the temperature started to drop, the furnace responded quite sluggishly when additional current was applied to the furnace coils. The reason for this sluggishness can undoubtedly be attributed to the fact that the furnace was operated at or near the maximum temperature obtainable when it was wired for 110 volts.

The time element proved to be the greatest hindrance in the conductance of the tests. Each individual reduction test required considerable time. A time period of about 1 1/2 hours was required for
the furnace to reach the operating temperature, and along with that
impedance, it was necessary to place the charge in the sillimanite
tube and place the tube in the furnace before the furnace started
to heat. Trouble was experienced with tube breakage if the furnace
was at operating temperature when the tube was inserted. The furnace
was very slow in cooling after a test was conducted, consequently, a
period of from 5 to 6 hours was required between tests if the tests
were conducted one after the other. No tests were conducted in which
a silica tube was used because the operating temperatures of the
furnace made a silica refractory almost prohibitive.

Nothing could be done to prevent the gas from channeling within
the tube, and only the actual surface of the charge exposed would be
effected by the gas reducing agent. Attempts to pressurize the gas
within the tube failed because of the system of rabbling which was
used and also, the discharge end of the tube had to be kept open so
that the pyrometer could be inserted. Because of the channeling of
the gas and the method of rabbling used, it was difficult to insure
equal exposure time to all particles in the charge, and thus, the
final product was undoubtedly affected by this condition. In the
same line, when the tube was rotated, the charge had a tendency
to level out, and particles of the charge would migrate into the cold
zones of the tube, and the only way they could be placed back in
the reduction zone was by ramming a rod into the end of the furnace.
This practice necessitated the removal of the gas line from one end
of the furnace, which, in turn, allowed an oxidizing condition to
exist in the furnace during the time the gas was disconnected.
Because of the size of the particles used to conduct these tests, the rate of gas flow was definitely limited, since the finer particles were picked up by the velocity of the flow and carried out of the tube if the flow became excessive. All of these factors tended to give evidence that some of the charge was, in the main, always out of the reducing atmosphere, and consequently, when the charge was removed from the tube, these unreduced particles tended to lower the results of the final-product assays and caused the process to show a lower efficiency of operation than could be accomplished.

When the process was started on the dead-roasted pyrites, and before the gas was admitted, the charge was very fluid, but when the reduction actually started and the mass started to assume its sponge-like appearance, the fluidity of the mass decreased quite rapidly, the rabbling of the charge became more difficult, and a constant exposure time to the entire mass more uncertain.
### DATA

**Pyrite Concentrate Assay**

- Fe: 37.4%
- S: 43.7%
- Insol: 15.4%

### Screen Analysis of Pyrite Concentrate

<table>
<thead>
<tr>
<th>Mesh</th>
<th>Per Cent (Wt.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+65</td>
<td>14.9</td>
</tr>
<tr>
<td>-65 +100</td>
<td>33.4</td>
</tr>
<tr>
<td>-100 +150</td>
<td>26.5</td>
</tr>
<tr>
<td>-150 +200</td>
<td>12.7</td>
</tr>
<tr>
<td>-200</td>
<td>12.5</td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
</tr>
</tbody>
</table>

### Preliminary Tests

Dead-roasted pyrite samples which weighed approximately 45 g were subjected to the reducing effects of the natural gas, at elevated temperatures, for a period of about one hour and then cooled in a flow of cool gas. After the charge had reached room temperature the product was placed in the magnetic field of a strong magnet and a majority of the particles behaved very much like metallic iron particles. When a sample of the product was placed in a copper-sulfate solution, cementation of the copper was rapid, and further proof that metallic iron particles were present.
Results of Tests with Dead-Roasted Pyrites

From the standardization of the potassium-dichromate solution, the iron titre was established at 0.01665.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Temp. (F)</th>
<th>Time (Hr)</th>
<th>Total Fe (g)</th>
<th>Sponge Fe (g)</th>
<th>Per Cent Reduction</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1600</td>
<td>1</td>
<td>0.1367</td>
<td>0.1132</td>
<td>82.95</td>
</tr>
<tr>
<td>2</td>
<td>1700</td>
<td>1</td>
<td>0.1214</td>
<td>0.1108</td>
<td>91.02</td>
</tr>
<tr>
<td>3</td>
<td>1800</td>
<td>1</td>
<td>0.0667</td>
<td>0.0633</td>
<td>94.70</td>
</tr>
<tr>
<td>4*</td>
<td>1600</td>
<td>2</td>
<td>0.0766</td>
<td>0.0550</td>
<td>71.74</td>
</tr>
<tr>
<td>5*</td>
<td>1700</td>
<td>2</td>
<td>0.0538</td>
<td>0.0450</td>
<td>83.55</td>
</tr>
<tr>
<td>6</td>
<td>1800</td>
<td>2</td>
<td>0.0748</td>
<td>0.0629</td>
<td>84.13</td>
</tr>
<tr>
<td>7</td>
<td>1700</td>
<td>2</td>
<td>0.0735</td>
<td>0.0675</td>
<td>91.95</td>
</tr>
<tr>
<td>8</td>
<td>1800</td>
<td>2</td>
<td>0.0698</td>
<td>0.0666</td>
<td>95.23</td>
</tr>
</tbody>
</table>

* Pyrometer Leads broken and test results erroneous
Results of tests with 6.3% Sulfur Content in Pyritic Charge to the Reduction Step

The charge was placed in the furnace which was operating between 800 and 900 F, and the gas was connected immediately. At a temperature of 1600 F, the charge was completely sintered and was no longer rabbled. A period of 1½ hours was required for the furnace to reach a temperature of 1800 F, after which, the temperature was held constant for another ½ hour.

The results of these tests were as follows:

<table>
<thead>
<tr>
<th>Sponge Iron (g)</th>
<th>Total Iron (g)</th>
<th>Per Cent Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0328</td>
<td>0.0683</td>
<td>48.10</td>
</tr>
</tbody>
</table>

A grab sample of the dead-roasted pyritic feed was taken and assayed and it showed a total-iron content of 32 per cent.
DISCUSSION OF RESULTS

From the data obtained, it was evident that the temperature was the important variable in the reduction procedure. The best recovery was obtained at a temperature of 1800 F and apparently the greatest reduction was obtained within the first hour since the products of the two-hour tests did not show an appreciable gain in metallic iron content when the time element was considered.

It is evident from the tests conducted, that the sponge iron can be obtained in two different forms. If a loose, granular end product is desired, the pyrite ore should be dead roasted prior to the reduction procedure, and if a sintered product is desired, the pyrite ore should not be dead roasted but subjected to the reduction step with a low-sulfur content.

When the cooling procedure used in the third type of tests was employed, a light, fluffy, coating of carbon was encountered on the sponge-iron sinter. This carbon coating did not adhere tenaciously to the surface of the final product and could be removed fairly easily by an air blast or by wiping. After the majority of the elemental carbon had been removed, the solution of the metal in acid was much more rapid than it had been previously when the reduced product was cooled in a flow of the gas.

When the non-sintered final product was pulverized for assaying, the badly-carburized samples proved very difficult to place in solution even after long periods of digestion; also, the problem of skin flotation of the fine, carbon-coated particles made it very difficult
to obtain complete dissolution of the material even when severe agitation and evaporation to dryness with aqua regia was practiced.

Since all the calculations were relatively simple, they have been omitted from the data sheets. The per cent reduction of the iron can be calculated easily, either by the direct calculation of the ratio of the milliliters of standard dichromate solution used for the sponge iron titrations to the milliliters of solution used for the total iron titrations, or by the method used on the data sheets, in which, the iron titre is multiplied by the milliliters used and the ratio obtained on the weight basis.

The pyrite concentrates which were used seemed readily adaptable to this type of experimentation. The silica content seemed high enough to prevent any incipient fusion during the roasting process, and when the sintered product was obtained in the reduction step, the porosity of the reduced particles was such that reduction took place into the very center of the piece. During the experimentation with the sintered cake, the flow of gas was greatly increased once the sintering had taken place. The flow of gas could be increased because the loss of fine particles to the flow of the gas was eliminated, and also the chance of a greater penetration of the gas into the charge without the necessity of continual rabbling was enhanced.

No attempt was made to measure the amount of gas used during the tests. The rate of gas flow was regulated by the length of flame which protruded from the end of the tube. This method was used to give a uniformity of test conditions for one of the dependent variables.
The gray sponge obtained in the sintered cake from the second method of cooling proved very amenable to the assay tests used. The material responded very readily to the hydrochloric acid solution and a 0.2500-g sample was easily dissolved in a matter of from 2 to 3 hours, while the badly carburized particles, from the previous tests, which were cooled under the reducing effect of the gas, sometimes required a period of from 2 to 3 days in order that complete dissolution could be assured.
CONCLUSIONS AND RECOMMENDATIONS

At elevated temperatures, sponge iron can be produced from roasted pyrites, when natural gas is used as the reducing agent. Metallic iron can be obtained from either a dead- or a low-sulfur roasted product. For the most rapid reduction rate, the minimum operating temperature is 1800 F, and at this temperature, any iron oxide apparently is reduced to the metallic state almost immediately. If a moderate degree of porosity exists in the charge, the gas seems to penetrate very well. In the small-scale experiments conducted for this paper, with the furnace operating at 1800 F for only ½ hour, (see Test 9) reduction was effected to the center of a piece which was 5/8 in. in diameter.

In a dead-roasted pyritic charge, reductions up to 95 per cent can be anticipated, and under ideal conditions, 100 per cent reduction should be achieved. The limitations of the sulfur content cannot be established through the tests conducted thus far, but metallic iron can be produced from a roasted product which contains up to 6 per cent sulfur.

The temperature of the reduction step is the most important variable in the process when only the time and temperature variables are considered. The optimum reducing conditions were obtained at 1800 F which was the highest temperature utilized during any of the tests. Since carbon monoxide cannot act as a reducing agent in the presence of carbon much above 1835 F, it is possible that the efficiency of the operation would decrease somewhat above this temperature because
of the elemental carbon which is produced during the reduction.\textsuperscript{5}

In order to obtain a product which is relatively carbon free, the discharge product should be cooled in air rather than under a strong reducing atmosphere. The detrimental effects of carbon occlusions are much greater than the degree of oxidation obtained in the final product. Very little oxidation of the final product was observed even after the metallic sponge stood in an air atmosphere for several days; and, if subsequent treatment were to follow the reduction step, ample time for this treatment should be available before any impedance could be brought about because of this oxide coating.

No methods of separating the metallic iron from the gangue and other impurities were attempted so no procedure for this process can be outlined at this time.

From the data obtained from the tests performed, it is possible that this process could be made to operate on a commercial basis. The fundamentals of the process are basically sound and if the right equipment were to be designed, it is feasible that a valuable by-product could be obtained from primary smelting operations where pyrites are roasted for their sulfur content for acid production, and where there is an ample supply of natural gas to operate a process of this nature. For example, if two fluo-solids reactors were used side by side, and the pyrites subjected to a fluidizing roast in one with the sulfur dioxide gas removed in the conventional manner at the top of the reactor; and the underflow or roasted product removed and introduced into the other while the charge was still hot.
and natural gas, with some external heat, supplied in a counter-
current manner, it is possible that metallic iron could be produced
in the second reactor and removed as the underflow; thus, a continuous
operation could be made of the process. A batch process might be
conducted in one reactor alone in which the air would be added at
the beginning in order to roast the charge and after the completion
of the roast, natural gas would be added to effect the reduction
of the iron oxide which remained in the charge to metallic sponge
iron.

A definite obstacle to these procedures would have to be
overcome in some manner before these operations would become success-
ful. At the beginning of the reduction step the charge is very
fluid but when a considerable amount of the reduction has taken
place, the fluidity of the charge is lost and the iron appears
to agglomerate and ceases to flow readily. Since also it is apparent
that a 6 per cent sulfur content caused a sintering of the product
at 1600 °F, an initial feed to the reduction step of this sulfur
percentage would prohibit its use in this type of process. However,
the sulfur percentages of sulfur
might prove successful. A dead-roasted product, with the restrictions
anticipated above, might be readily amenable to the above-described
process.

If an agglomerated product were desired from a fine initial
product, a partially-roasted product introduced into a rotary kiln
through which natural gas was passed might give a product which could
be satisfactorily used in the cementation of copper from copper-
bearing solutions. These larger agglomerates would be more desirable
in the cementation tanks to alleviate the loss of values through slime losses and greatly aid the settling problem encountered in thickeners.

The recommendations may be summarized as follows:

1. Since the results of the experimentation show that sponge iron can be produced from pyrites, with a natural gas reduction, further work should be done to obtain the best conditions under which the process will operate.

2. Laboratory equipment should be devised to approximate a fluidizing roast so the heat of the exothermic roast reaction can be utilized in the subsequent reduction step.

3. More exhaustive tests in which varying percentages of sulfur are used should be conducted to find the effects of sulfur content on both, the physical characteristics of the final reduced product, and also, on the effect the sulfur has on the reduction process with regards to time, temperature, and degree of reduction.

4. Different sizes of the pyrite particles should be subjected to these tests in an endeavor to establish the mesh size which gives the best results.

5. Equipment which lends itself to more accurate quantitative results should be employed in further tests.

6. The data obtained in these tests should be considered as strong indications rather than exact facts, as to what can be expected under the circumstances described, since the equipment used in these tests was not designed to obtain
quantitative results.

7. The gas consumption should be measured in some manner so that the process can be placed on a dollar-and-cent basis.

8. Very serious consideration should be given the aspects of this process for their adaptation to a commercial process for the recovery of an important metal which has heretofore been entirely wasted.

9. The sponge iron product should be subjected to flotation tests in an endeavor to obtain a high-grade, metallic iron concentrate because the iron particles should be very pure since the temperatures reached in this method of metallic iron production are low compared to the melting point of iron and, consequently, the solution of impurities in the iron should be nil.
ACKNOWLEDGEMENT

For their valuable assistance rendered to me in the furtherance of my work described in this paper, I wish to thank Dr. J. G. Grunenfelder, Professor R. I. Smith, and Dr. Kenneth McLeod.


OTHER REFERENCES


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