RI 7627

Bureau of Mines Report of Investigations/ 1972

Flash Reduction of Iron Ore

UNITED STATES DEPARTMENT OF THE INTERIOR

Report of Investigations 7627

Flash Reduction of Iron Ore

By E. G. Davis and I. L. Feld
Tuscaloosa Metallurgy Research Laboratory, Tuscaloosa, Ala.

UNITED STATES DEPARTMENT OF THE INTERIOR
Rogers C. B. Morton, Secretary

BUREAU OF MINES
Elburt F. Osborn, Director

The work upon which this report is based was done under a cooperative agreement between the Bureau of Mines, U.S. Department of the Interior, and the University of Alabama.

This publication has been cataloged as follows:

Davis, Edward G

Flash reduction of iron ore, by E. G. Davis and I. L. Feld. [Washington] U.S. Dept. of the Interior, Bureau of Mines [1972]

10 p. illus., tables. (U.S. Bureau of Mines. Report of investigations 7627)

Based on work done in cooperation with the University of Alabama.

1. Iron ores. 2. Reduction, Chemical. I. Feld, Ignatz L. II. Title. (Series)

TN23.U7 no. 7627 622.06173

U.S. Dept. of the Int. Library

CONTENTS

ILLUSTRATION

TABLES

FLASH REDUCTION OF IRON ORE

by

E. G. Davis[1] and I. L. Feld[2]

ABSTRACT

The flash reduction of fine iron ore in a dilute-phase system was investigated at 500° to 900° C within a heated stainless steel tubular coil. Alabama brown iron ore and Mesabi semitaconite ore were fed into the coil concurrently with reducing gases of hydrogen and reformed natural gas, so that the velocity of the gas at the reaction temperature was sufficient to entrain and pneumatically transport the solids through the coil.

Reduction is very rapid with retention time of the ore in the coil varying from 4 to 15 seconds. Variables affecting reduction are temperature, ore feed rate, and gas flow or velocity.

Results of the studies show that a partially reduced product (10- to 25-percent reduction) can be made which, on subsequent magnetic concentration, yields a concentrate containing 64 to 70 percent iron with a 90- to 94-percent iron recovery. Maximum reduction of 77 percent was obtained when hydrogen was used in a four-stage reduction test to yield a product containing 61 percent metallic iron.

INTRODUCTION

Various methods for treating low-grade iron ores to remove oxygen are in use today. The reduction of iron oxide by reduction roasting is one method that has received considerable attention in recent years. Reduction roasting of hematitic iron ore may result in either a highly reduced product (metallized), or a partially reduced product (magnetized). The reduced product is then concentrated by magnetic separation techniques to yield a high iron concentrate. The metallized iron concentrate can be used as feed to electric furnaces for the production of steel, or in the foundry industry for the production of cast iron or ductile iron products. The magnetic iron concentrate can be used as a feed to the blast furnace for the production of pig iron.

---

[1]Metallurgist.
[2]Supervisory metallurgist.

Several methods for reduction roasting have been proposed, some of which are commercially attractive. These include the shaft furnace and rotary kiln methods in which solid or gaseous reductants are used, and the fluidized-bed method in which gaseous reductants are used. The effective treatment of fine particles in the ore is a problem in all these methods. In each case, especially the fluidized-bed, special dust collecting and recycling systems are required to prevent excessive loss of the fine particles. Because of these problems with the fine-sized material, the possibility of reducing fine-sized iron ore in a flash type reactor was investigated. In this method, the fine iron ore is entrained and pneumatically transported through the reactor at the desired temperature, and flash reduced in a very short period of time.

The proposed method is based in part on previous work done by the Bureau on extracting oil from oil shale by using an entrained-solids retort.[3] In this method the oil was extracted from the pulverized shale in a very short period of time by reacting it with steam while the shale was entrained, and pneumatically transported through the coil reactor at the reaction temperature.

This report covers the construction and performance of a coil reactor for the flash reduction of fine iron ore with hydrogen and oxygen-reformed natural gas as the reductants.

CONSTRUCTION OF COIL REDUCTION SYSTEM

The coil reduction system consisted of the coil, feeder, gas preheater, furnace, collection flask, and gas-flowmeter as shown in figure 1.

For this investigation, two coils were constructed. The first coil was made from 20 feet of 1/2-inch, type 347 stainless steel tubing, having an ID of 0.467 inches and a coil diameter of 9 inches. The coil was placed in the furnace so that 16 feet of the tube was in the furnace hot zone. The second coil was identical to the first coil except that a 40-foot length of tube was employed so that 32 feet would be in the furnace hot zone. By doubling the length of the coil in the hot zone, the retention time during reduction would be increased thus providing a way to determine whether retention time affects the total reduction.

The feeder was constructed with a hopper, a horizontal screw, and a gas-ore mixing chamber so that the ore could be fed into the gas stream. The feed rate was controlled by adjusting the speed of a variable speed motor used to turn the screw. A sealed hopper feed entry and a gas equalization line to the hopper was used to prevent extraneous gases from entering the hopper and upsetting the solids feed.

A gas preheater was used to insure that the initial gas flow had sufficient velocity to transport the ore from the feeder to the coil in the furnace hot zone. It was constructed from a 1-inch stainless steel pipe containing

---

[3] Sohns, H. W., E. E. Jukkola, and W. I. R. Murphy. Development and Operation of an Experimental, Entrained-Solids, Oil-Shale Retort. BuMines Rept. of Inv. 5522, 1959, 45 pp.

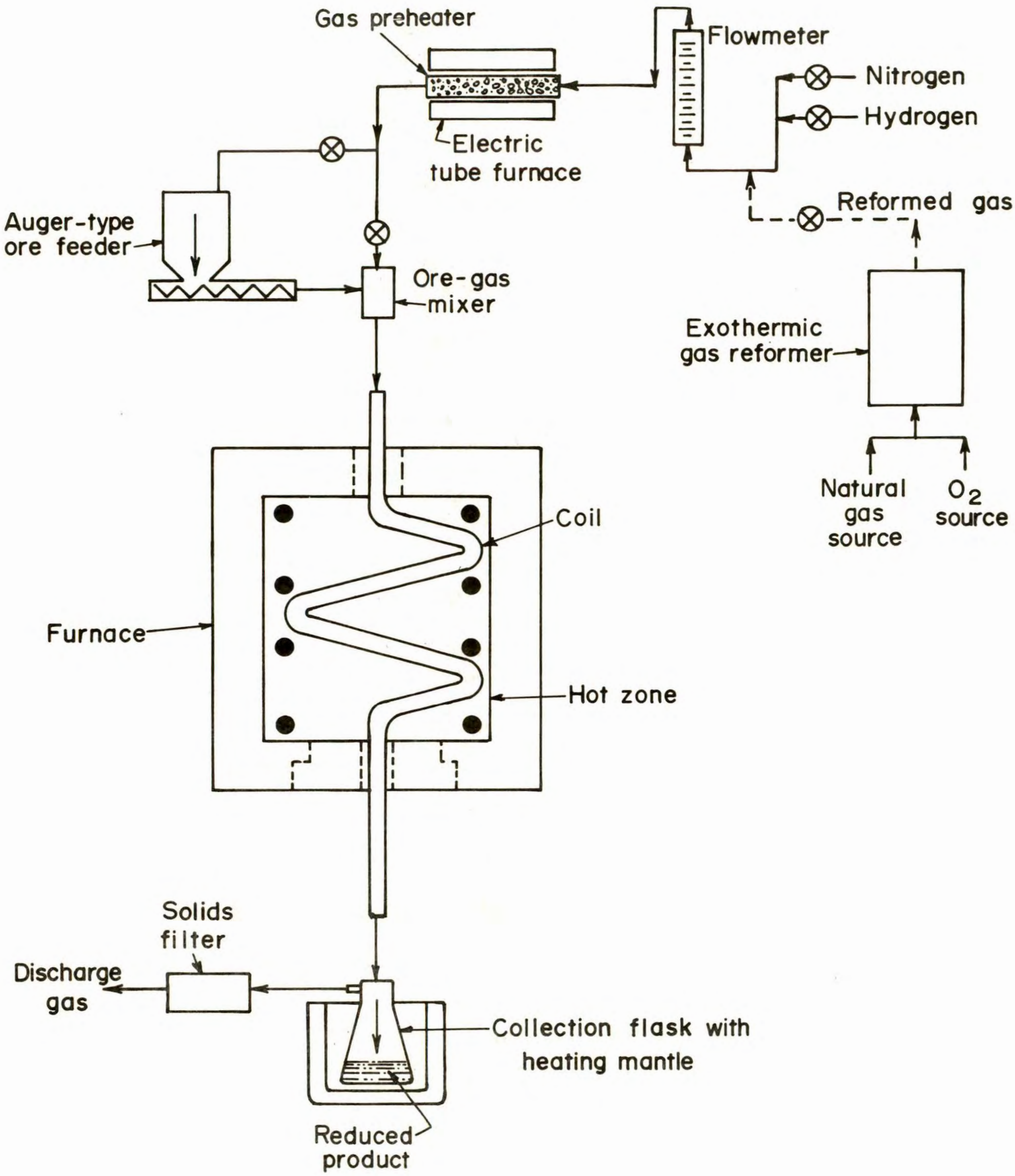

FIGURE 1. - Schematic Drawing of Coil Reduction System.

nickel shot as the heat transfer material. A small tube furnace was used to supply the heat to the gas preheater.

The solids collector was a vacuum flask fitted to the end of the tubing with a silicone rubber stopper. A heating mantle was placed around the flask to prevent the water vapor that formed during the reduction process from condensing on the reduced solids. The discharge gas from the collection flask was passed through a filter to remove all solid material.

MATERIALS USED

Samples of limonitic brown iron ore from southeastern Alabama and samples of a nonmagnetic semitaconite ore from the western Mesabi range in Minnesota were used in this study. Both are low-grade "earthy" iron ores containing a considerable amount of fine particles; however, to insure a uniform size feed, both ores were ground to pass 100-, 200-, or 400-mesh size depending on the test conditions. Analysis of both ores is shown in table 1.

TABLE 1. - Chemical analysis of iron ores used in experiment

Ore	Analysis, weight-percent				
	Fe	SiO_2	Al_2O_3	P	Loss on ignition[1]
Alabama brown ore............	47.3	10.9	5.1	0.42	13.5
Minnesota semitaconite........	43.2	32.8	.9	.03	4.0

[1] Ignition made at 1,000° C.

The reducing gases used in this study were hydrogen and an oxygen-reformed natural gas containing 55 percent H_2, 31 percent CO, 10 percent CO_2, 1 percent N_2, and 3 percent H_2O. The reformed gas was produced from oxygen and natural gas in an exothermic reformer. The oxygen used in the reformer, the hydrogen used for reduction, and the nitrogen used for purge gas were of commercial purity.

EXPERIMENTAL PROCEDURE

The procedure for all tests made in the coil reactor was the same. Nitrogen was first passed through the coil until the desired temperature was obtained. The reducing gas was then introduced. When the desired velocity through the coil at the operating temperature, as predetermined by flow rate, was maintained, the nitrogen was discontinued. The fine ore was then fed into the preheated gas stream at a predetermined rate using the rotational speed of the screw for controlling the feed rate. The reduced ore was collected in the collection flask. At the end of each test, the feeder was stopped, the reducing gas discontinued, the system purged with nitrogen, and the collection flask was removed from the coil and heating mantle.

The top of the flask and its gas discharge line were stoppered to prevent reoxidation of the product during cooling to room temperature prior to removing for analysis. In some tests a water quench tank was used instead of the collection flask. In these tests, the procedure was identical except for collecting the product in the quenching tank where the reduced sample was instantaneously cooled to prevent reoxidation. After collection the product was filtered and dried prior to analysis.

The percent reduction shown in all test data is calculated on the basis of oxygen loss from iron oxide.

A Davis tube magnetic separator was used with a standardized separating technique in all magnetic concentration tests.

RESULTS

Metallizing

As in fluid-bed reduction studies,[4] the reduction in the coil at 750° to 900° C is determined by the mole ratio of the amount of reductant in the gas per unit time to the amount of oxygen as iron oxide in the corresponding quantity of entrained ore. Unlike the reduction of coarser particles in the fluid-bed method of Hansen's report, the fineness of particles in the feed in the entrained coil reactor should accelerate the reduction rate, thereby attaining a high degree of reduction in an extremely short period of time, while the particle is transported through the coil.

Preliminary tests using the coil reactor indicated that three variables affect reduction. These variables are (1) temperature, (2) ore feed rate, and (3) gas flow rate or velocity. Preliminary tests also showed that a minimum velocity of 50 feet per second at temperature must be used to transport the solids through the coil and that the velocity is the primary factor for determining the solids retention time.

A series of tests was designed using the Latin Square[5] technique for studying these three variables to determine the best conditions for obtaining a highly reduced product. Iron ore reduction studies[6] have shown that to obtain a high level of reduction at rapid reduction rates, the reaction temperature should be above 750° C with best results being obtained at 900° C. As a result of this earlier work, the temperatures used in this series of tests were 750°, 825°, and 900° C. Also, caking of the ore and sagging of the coil became serious problems at temperatures above 900° C. Feed rates of 18, 36, and 54 grams per minute, were used for these tests. These rates correspond to 1, 2, and 3 tons of ore being processed per square foot of coil cross-sectional area per hour. Velocities of 60, 90, and 120 feet per second were chosen, all above the minimum velocity of 50 feet per second. The ratio of moles of hydrogen in the gas to moles of oxygen in the iron oxide in the ore varied from 8.6 to 50. Results of these tests made on minus 200-mesh Alabama brown ore using hydrogen as the reductant are shown in table 2. A statistical analysis of these results showed that the optimum test conditions for producing the highest reduction were (1) 900° C reaction temperature, (2) 18 grams per minute feed rate, and (3) 120 feet per second gas velocity or 2 cubic feet per minute STP gas flow rate. The mole ratio of reductant to oxygen in

---

[4]Hansen, J. P., J. E. Berryhill, and J. A. Aufman. Batch Reduction of Iron Ore in Fluidized Bed. BuMines Rept. of Inv. 7461, 1970, 25 pp.

[5]Natrella, Mary G. Experimental Statistics. National Bureau of Standards Handbook 91, Aug. 1, 1963, pp. 13·30-13·35.

[6]Work cited in footnote 4.

iron oxide is 43 at these conditions. The retention time of the solids in the coil varied between 4 and 10 seconds for all tests. A test made with these optimum conditions resulted in a 53-percent reduced product.

TABLE 2. - Reduction of minus 200-mesh Alabama brown ore[1]

Test	Coil temperature, ° C	Ore feed rate		Gas		Iron analysis of product, weight-percent				Reduction, percent
		G/min	Ton/ft² /hr	Flow, ft³/min (STP)	Velocity, ft/sec at temperature	Fe, metallic	Fe^{++}	Fe^{+++}	Fe, total	
1	750	54	3	1.2	60	0.5	22.5	33.2	56.2	14.3
2	750	36	2	1.7	90	2.3	34.9	19.4	56.6	25.2
3	750	18	1	2.3	120	7.4	31.9	21.6	60.9	29.9
4	825	54	3	1.6	90	Trace	35.2	22.4	57.6	20.5
5	825	36	2	2.2	120	13.1	45.5	-	58.6	48.6
6	825	18	1	1.1	60	5.3	43.8	9.4	58.5	34.1
7	900	54	3	2.0	120	3.7	47.8	8.6	60.1	32.7
8	900	36	2	1.0	60	5.3	47.4	7.0	59.7	35.5
9	900	18	1	1.5	90	20.0	36.4	5.7	62.1	51.8

[1] 16 feet of coil in the hot zone with hydrogen used as the reductant.

To obtain a higher reduced product, a test was made using hydrogen as the reductant whereby the product from the first pass was recycled through the coil three additional times thus simulating a 4-stage reduction operation. The temperature, feed rate, and gas velocity for all four runs or stages were 900° C, 18 grams per minute, and 60 feet per second, respectively. Results of this 4-stage test, as shown in table 3, indicate that a maximum reduction of 77 percent was obtained in a product containing 61 percent metallic iron after four passes.

TABLE 3. - Staged reduction of minus 200-mesh Alabama brown iron ore[1]

Test	Stage	Reduction, percent	
		Each stage	Cumulative
1	1	34.9	34.9
2	2	20.6	55.5
3	3	9.1	64.6
4	4	12.4	77.0

[1] 900° C, 18-g/min feed, 60-ft/sec gas velocity each stage, 16 feet of coil in hot zone with hydrogen used as the reductant.

Results of these metallizing tests were not encouraging because of the limited reduction obtained. Furthermore, a preliminary economic evaluation of the process given in an unpublished report by the Bureau's economic evaluation group at the College Park Metallurgy Research Center indicated that the process was not commercially attractive for producing a metallized product.

Magnetizing

The performance of the flash reduction method also was evaluated for achieving partial reduction of the low-grade iron ores to yield magnetic iron oxide. Subsequent magnetic concentration of this product could be used to obtain high-grade iron oxide concentrates that would be suitable for use as a blast furnace feed.

Reduction studies on iron ore have shown that a reduction of 10 to 25 percent gives a product that can be magnetically separated to yield a concentrate of 64 to 70 percent iron with a 90- to 95-percent iron recovery. Theoretically, according to the formula,

$$3Fe_2O_3 + H_2 \rightarrow 2Fe_3O_4 + H_2O, \qquad (1)$$

11 percent of the total oxygen is removed, and the iron in the reduced product is Fe_3O_4.

A series of tests designed according to the Latin Square technique was therefore made on minus 100-mesh semitaconite ore. Hydrogen was used as the reductant to determine the best conditions for producing a partially reduced product. The results are shown in table 4.

TABLE 4. - Results of reducing minus 100-mesh semitaconite ore[1]

Test	Coil temperature, ° C	Ore feed rate, g/min	Gas velocity, ft/sec	Iron analysis of product, weight-percent				Reduction, percent	Magnetic concentrate, weight-percent	
				Fe, metallic	Fe^{++}	Fe^{+++}	Fe, total		Fe	Fe recovery
1	500	60	110	0.8	5.7	38.5	45.0	6.0	66.3	57
2	500	40	80	.8	5.7	38.9	45.4	6.0	67.1	37
3	500	20	50	.8	5.7	37.7	44.2	6.1	65.1	63
4	600	60	80	2.0	15.4	29.2	46.6	14.7	68.1	81
5	600	40	50	7.6	14.6	28.1	50.3	24.8	67.2	88
6	600	20	110	2.0	14.6	30.0	46.6	14.6	68.1	83
7	700	60	50	.4	17.0	26.8	44.2	13.8	65.7	94
8	700	40	110	2.4	18.7	25.1	46.2	18.7	68.9	90
9	700	20	80	6.1	25.5	15.4	47.0	31.0	71.0	90

[1]16 feet of coil in hot zone, and hydrogen used as the reductant.

The temperatures used were lower than in the previous test because reduction studies made in the fluid-bed have shown that partial reduction can be accomplished at temperatures between 500° and 700° C. The other two variables, ore feed rate and gas velocity, were the same as in the previous tests. Statistical analysis of the reduction tests based on percentage reduction showed that the optimum conditions for producing the least reduction, while still remaining in the range of 10 to 25 percent, were a temperature of 700° C and a feed of 60 grams per minute with a gas velocity of 110 feet per second.

The magnetic concentrates from all tests, also shown in table 4, contained at least 65 percent iron, but the recovery varied. Therefore, a statistical analysis was made of these tests to determine the best conditions for obtaining a high iron recovery in the magnetic concentrate. The best conditions were also a temperature of 700° C and a feed of 60 grams per minute but with a gas velocity of 50 feet per second.

Further evaluation of both statistical analyses showed that the values obtained for gas velocity of 80 feet per second were fairly close to the optimum selected in both cases. Because this variable was the only difference and it was the mean velocity of the two statistical analyses, a test using a temperature of 700° C, a feed rate of 60 grams per minute, and a gas velocity of 80 feet per second was made to determine whether this gas velocity would give the desired product. The results of this test showed that an 11 percent reduced product, which yielded a magnetic product analyzing 65 percent iron with a 94-percent iron recovery, was made.

Additional tests on minus 100- and minus 400-mesh semitaconite ore using a temperature of 700° C were made to determine the effect of particle size and gas-ore ratio on the reduction percent. Magnetic separation tests were also made to determine the type of concentrate available from the reduced products.

Results of these tests are presented in table 5. Because the reduction rate of Fe_2O_3 to Fe_3O_4 is very rapid at these test conditions and the retention time was from 4 to 10 seconds, the effect of particle size and gas-ore ratio was inconclusive. The only conclusive result is that a reduction between 10 and 25 percent produces a reduced product which yields a high-grade magnetic concentrate with good iron recovery.

TABLE 5. - Reduction and concentration of semitaconite ore[1]

Test	Ore		Gas velocity, ft/sec	Reduction, percent	Magnetic concentrate, weight-percent	
	Size, mesh	Feed rate, g/min			Fe	Fe recovery
1	Minus 100	20	80	31.0	71.0	90.0
2	...do.....	30	80	18.3	67.5	95.0
3	...do.....	40	110	18.7	68.9	90.0
4	...do.....	60	50	10.4	65.3	97.0
5	...do.....	60	80	13.3	65.3	93.0
6	Minus 400	60	50	21.6	70.0	90.0
7	...do.....	20	80	22.6	70.1	91.0
8	...do.....	40	110	22.4	70.0	90.0
9	...do.....	20	80	21.8	70.3	93.4
10	...do.....	60	80	11.9	67.5	92.6

[1] 16 feet of coil in hot zone, at 700° C with hydrogen used as the reductant.

Tests were made at 700° C, in the coil of which 32 feet (rather than 16 feet) were inside the furnace hot zone. The gas velocity was 80 feet per second and the feed rates varied from 20 to 60 grams per minute. The ores used were minus 200-mesh Alabama brown ore and minus 100- and 400-mesh semitaconite with hydrogen as the reducing gas. The results are shown in table 6. The retention time in these tests was from 10 to 15 seconds, indicating that doubling the length of tubing in the hot zone only increased the solids retention time by about 1.5 to 2. The percent reduction obtained on the semitaconite ore was somewhat higher than results with the shorter coil. The grade of magnetic concentrates was about the same as that of products from tests made with the shorter coil, but iron recovery was lower in some cases probably owing to greater reduction. Results in the table also show that reduction of the brown iron ore was not as great as that for the semitaconites. Nevertheless the magnetic concentrates were of high grade and good recovery of iron was obtained.

TABLE 6. - Reduction of iron ore with hydrogen in the long coil[1]

Test	Ore	Feed rate, g/min	Reduction, percent	Magnetic concentrate, weight-percent	
				Fe	Fe recovery
1	Alabama brown ore, minus 200 mesh.	20	26.0	67.6	86.0
2	...do.............	40	17.7	65.2	92.0
3	...do.............	60	14.7	64.1	91.0
4	Semitaconite, minus 100 mesh.	20	13.3	67.4	90.1
5	...do.............	40	15.1	64.0	92.1
6	...do.............	60	14.5	66.1	91.0
7	Semitaconite, minus 400 mesh.	20	37.9	65.0	88.0
8	...do.............	40	29.2	67.9	89.2
9	...do.............	60	21.5	68.0	91.0

[1] 32 feet of coil in hot zone, at 700° C, 80 ft/sec gas velocity.

Comparative tests were made on both ores using oxygen-reformed natural gas instead of hydrogen as reductant. The results, given in table 7, generally indicated that percent reduction was greater with hydrogen. Because of the lesser reduction obtained with the reformed gas, magnetic concentrates from these reduced products were lower in grade, but iron recovery was increased.

TABLE 7. - Reduction of iron ore with oxygen-reformed natural gas in the long coil[1]

Test	Ore	Feed rate, g/min	Reduction, percent	Magnetic concentrate, weight-percent	
				Fe	Fe recovery
1	Alabama brown ore, minus 200 mesh.	20	10.1	64.0	91.0
2	...do.............	40	9.6	62.6	94.8
3	...do.............	60	9.0	62.0	92.1
4	Semitaconite, minus 400 mesh.	20	17.4	66.9	93.5
5	...do.............	40	16.9	67.5	92.4
6	...do.............	60	24.5	66.5	90.1

[1] 32 feet of coil in hot zone, at 700° C, 80 ft/sec gas velocity.

In many tests throughout this investigation the reduced products were pyrophoric, resulting in a handling and analyzing problem even after the products were cooled under a nitrogen atmosphere. The products were always handled and analyzed under an inert atmosphere to insure that no reoxidation occurred.

Tests were made wherein the reduced product was water quenched immediately after reduction in order to reduce or eliminate the pyrophoric problem. Water quenching was performed by inserting the discharge end of the coil directly into the quench tank. Results of these tests, given in table 8, show that after quenching, filtering, and drying at 110° C some reoxidation did occur. However, none of the samples treated in this manner were pyrophoric and good magnetic concentrates were obtained in all cases.

TABLE 8. - Results of water quenching the reduced products[1]

Test	Ore	Reducing gas	Feed rate, g/min	Reduction, percent	Magnetic concentrate, weight-percent	
					Fe	Fe recovery
1	Alabama brown ore, minus 200 mesh.	Reformed natural gas	20	8.0	66.7	94.9
2	...do.............	...do.......	40	8.3	66.5	95.2
3	...do.............	...do.......	60	8.7	66.3	96.6
4	Semitaconite, minus 100 mesh.	H_2..........	20	17.4	64.6	90.2
5	...do.............	H_2..........	40	14.4	66.4	91.8
6	...do.............	H_2..........	60	14.6	66.2	92.6
7	Semitaconite, minus 400 mesh.	Reformed natural gas	20	11.4	67.1	94.6
8	...do.............	...do.......	40	12.3	66.5	95.1
9	...do.............	...do.......	60	12.4	67.0	93.1

[1] 32 feet of coil in hot zone, 700° C, 80 ft/sec gas velocity.

CONCLUSIONS

1. Highly reduced products (90 to 95 percent reduction) were not obtained by flash reduction of fine iron ore in the coil reactor under the conditions specified.

2. Partially reduced products (10 to 25 percent reduction) were made by flash reduction of fine iron ore in the coil reactor which, upon subsequent magnetic concentration, yielded a concentrate containing 64 to 70 percent iron with a 90- to 95-percent iron recovery.

3. It is technically feasible to rapidly reduce fine low-grade iron ore in the coil reactor with a reducing gas to form a reduced product which can be concentrated magnetically to yield a high iron content. This product would be suitable for pelletizing for use as a blast furnace feed.

INT.-BU.OF MINES,PGH.,PA. 17402

U.S. GOVERNMENT PRINTING OFFICE: 1972—707-439:314