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Metallization of the Oxide of Iron in Ilmenite.

by
~~(X)~~ R. J. Traill & ~~(X)~~ W. R. McClelland. ^x

~~(1)~~ Engineer, Mines Branch, Dept of Mines, Ottawa, Can.

~~(X)~~ Metallurgical Engineer, Mines Branch, Dept of Mines, Ottawa, Can.

Introduction.

Canada, has, in the provinces of Quebec and Ontario numerous deposits of ilmenite and titaniferous magnetite extensive in size and of good grade. No serious effort has been made to utilize these ores on a large scale. From time to time small shipments of these ores have been made to the United States, for the manufacture of ferro-titanium and titanium white. With the advent of extensive hydro-electric development affording cheap power, an interest has, however, been aroused in the problem of their reduction and utilization.

The investigation described in this paper deals with the metallizing of the oxide iron in Ilmenite in a rotary kiln furnace of semi-commercial proportions, based on work carried out previously in this laboratory. In the Report of Investigations of the Ore Dressing and Metallurgical Division, 1925, a report of this work is given outlining a method for the treatment of ilmenite ore, whereby the iron was separated from the titanium, resulting in the recovery of a high grade titanium dioxide concentrate suitable for the manufacture of titanium white pigment, and the recovery as a by-product of electrolytic iron.

Briefly the process consists in treating the crushed ore mixed with a suitable proportion of crushed coal and coke in a rotary kiln with exclusion of excess air at a temperature around 1000°C. The iron content is reduced to metallic iron in the form of a sponge, while the titanium content remains unaltered. By magnetic separation the unconsumed coke and coal, with some ore gangue are removed leaving a magnetic concentrate, containing the iron and titania. }

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To separate the iron and titanium two methods are applicable, solution of the iron either by ferric chloride or sulphuric acid. Where the recovery of iron is desired, the ferric chloride solvent is used, ferrous chloride being formed, and the iron recovered by electro-deposition in an electrolytic diaphragm cell, with concurrent regeneration of ferric chloride solvent. When the recovery of iron is not desired sulphuric acid, obtained from a subsequent stage in the treatment of titanium dioxide may be used as a solvent for the iron. The resulting ferrous sulphate is run to waste. By either method a high grade concentrate of titanium dioxide low in iron is obtained.

At the request of commercial interests the semi-commercial scale operations on the metallizing of the iron content of ilmenite were carried out early in 1928.

Description of Equipment.

The kiln type of furnace used was part of the equipment of the laboratories installed for the purpose of making investigations in sponge iron production from Canadian iron ores. This kiln is of the two-diameter type, ^{similar to that used} ~~as used and recommended~~ in the experimental work of the U.S. Bureau of Mines on sponge iron production and as reported in U. S. Dept of Commerce, Bureau of Mines Bulletin No.270, 1927. The kiln installed in the Ottawa Laboratory consisted of a steel shell lined with fire brick, having the following dimensions: Length of large section 5'6", inside diameter 2'6": One section tapering from diameter of 2'6" to 1'3" about 4' long, and connecting to the feed end section, 12' long and 1'3" diameter. The kiln rotated on two steel tires. The large end of the furnace was closed with a reinforced lining cement block and had a seven inch circular opening in the centre to permit of direct heating by means of an Anthony Nebulyte oil burner, type L-1 $\frac{1}{2}$. A circular combustion block, 13" in diameter, 9" long, and having a tapered combustion chamber opening from 3" to 7" diameter, was employed.

The block was suspended from above on rails thus permitting its easy removal when temperature readings were to be taken. With the block riding close to the furnace end, entrance of excess air was fairly well prevented.

A discharge pipe was inserted in the end wall of the furnace which fed into a hopper and thence to a bin. Precaution was made against undue oxidation as far as possible in the discharge arrangements. The feed arrangement consisted of a rectangular type hopper with a moving belt serving as the bottom and conveying the mix through an adjustable opening at end of hopper into a feed hopper to which was attached a 2½" diameter pipe leading some 8" inside the small end of the kiln. This end of the kiln opened into a dust chamber built of brick and having a baffle plate, the products of combustion thence passing on to the stack.

Experimental Tests.

The ore used in the tests was obtained from the deposit at Ivry, Terrebonne, Co. Que. and consisted of a hard medium to coarse grained ilmenite with some showings of hematite and of the following analysis:

Total iron (Fe)	39.00%
Titanium dioxide (TiO ₂)	31.75
Alumina (Al ₂ O ₃)	7.70
Lime (CaO)	0.45
Magnesia (MgO)	2.63
Silica (SiO ₂)	7.07
Sulphur (S)	0.73

Weight per cubic foot -8 mesh ore - 185 to 192 lbs. The coal used was obtained from a supply on hand at the Fuel Testing Laboratory of Alberta sub-bituminous of the following proximate analysis:

Moisture	9.00%
Ash	9.30
Volatile matter	34.10
Fixed carbon	47.60
Sulphur	0.45
Gross Calorific value	11,100 BTU

The coke used was a gas coke purchased locally and of the following approximate analysis (air dried basis):

Moisture	0.8%
Ash	13.1
Volatile matter	1.9
Fixed carbon	84.2
Sulphur	1.1
Gross calorific value	12,000 BTU

The fuel oil used was of ordinary market grade purchased locally.

The ore was crushed to pass an 8 mesh screen, the coal and coke to pass 4 mesh. In the initial tests the mix consisted of ore and coal only, from which there resulted a product low in metallization, and it was thought that this was perhaps due to the low fixed carbon content of the mix, and so it was decided to replace some of the coal with coke. The size of the discharge opening also occasioned some trouble in that too large an opening resulted in the mix passing through the furnace at too great a rate with a resulting low metallization. Various diameters were tried between $1\frac{1}{2}$ " and 1" and finally 1-1/8" discharge opening was adopted as a safe maximum. The feed mix consisted of ore and reducing material in the ratio of 5 to 4 and was mixed in batches by hand labour in the following proportions:

600 lbs.	Ore
320 lbs.	Coal
160 lbs.	Coke

The operating procedure was briefly as follows:

The discharge opening was cemented over, the furnace temperature raised to around 1000°C and 300 lbs. of mix charged into the wide diameter and through the opening in end wall of furnace, and a further 200 lbs. was fed in through the hopper. This charge was sufficient to give a good bed of material in the furnace which condition is very necessary to the successful operation of the furnace. When the temperature in the furnace ~~has~~ reached 990°C this temperature was maintained for one to two hours before the discharge pipe was opened. About 20 minutes previous to opening the discharge, the feed was started and while the furnace was discharging the feed was so regulated that a bed of sufficient depth was maintained in the furnace to keep the top level with the burner opening in the end of the furnace. This proved an important factor in the operation of the furnace as when the depth of charge lessened, the rate of discharge and the percentage of metallization fell off quite noticeably. Temperature also proved to be an important factor

and from the tests made it was found that a range of from 990° to 1040° C gave best results. Below 990° C reduction falls off and above 1040° C the effect tends to produce sintering and sticking of fines to the lining of the furnace.

Several preliminary runs averaging about 12 hours operation were made before sufficient data relative to the mechanical and operating conditions were obtained to warrant continuous operation. Two tests of the continuous type were run, the operating time being 100 and 104 hours respectively.

The continuous tests were run on the three shift basis. Each shift was responsible for mixing up the charge and for magnetic separation of product and sampling in addition to the regular furnace attention.

The discharge from the furnace was collected in covered bins and was removed every two hours, weighed and allowed to cool several hours before magnetic separation. Magnetic separation was made on a Gröndal Magnetic Belt Cobber at a belt speed of 300 ft. per minute and with 22 to 24 amperes on the drum and 8 amperes on the rectifier magnets. This machine gave three products, namely, a magnetic concentrate, a slightly magnetic middling, and a non-magnetic tailing. The analysis of these products are tabulated later. In plant operation the middling product would probably be returned with initial feed, but in the present tests it was not returned. The magnetic product from the Cobber was riffled in a Jones riffle sampler and the resulting sample ground in a Braun pulverizer and analysed for the metallic iron. The method of analysis used for the metallic iron determination was the well know copper sulphate method.

Continuous Test No. 1

Following the general procedure outlined above, a continuous test was run lasting 100 hours. Initial charge consisted of the regular mix as mentioned above, the temperature being held for one hour after rising to the desired temperature namely 990° C before the discharge was opened. The reduction temperature within the furnace was kept as closely as possible within 990°

and 1016 c. during the whole run. The following table shows the daily record of feed, discharge, concentration, per cent metallic iron, and the oil consumption:

TABLE 1

Hrs.	Feed lbs.	Feed/ hour lbs.	Disch lbs.	Disch/ hour lbs.	Mag. lbs.	Midd. lbs.	Non-mag. lbs.	% Met Fe		Oil consumed gals.
								Low	High	
24	2550	106.2	1776	74	972	276	534.5	30.54	41.46	45
24	2400	100	1597	66.5	924.5	128	541	39.06	43.12	40
24	2550	106.2	1626	67.7	735	279.5	537	26.39	44.78	35
24	2550	106.2	1691.5	70.5	1006.5	85.5	589.5	41.66	44.16	35
4	250		287.5	71.8	173	13	96	43.43	43.43	
100	10300	103	6978	69.78	3866	782	2298			155

During the first fourteen hours operation, some difficulty was experienced with the burner and with maintaining the proper depth of charge in the furnace due to feeder trouble with consequent irregular low metallization. Later, on the third day, a drop in metallization occurred but this was finally traced to an error in mixing the charge, in that the necessary proportion of coke had been omitted. During the rest of the run, conditions were normal and the results were quite satisfactory. The test yielding 2,945 pounds of satisfactory product, showing a metallization of 90-95%. Time did not permit determining total iron and titanium dioxide on all samples, but several samples taken at random showed the following analysis:

Met. Fe %	Total Fe %	TiO ₂ %	Metallization %
41.61	45.28	39.49	92.1
42.67	45.86	39.73	93.0
42.88	45.96	39.39	93.4
43.03	44.98	39.98	95.8
43.35	46.15	40.07	93.9

The discharge from the furnace has a weight per cu.ft. of 77-78 lbs and the magnetic concentrate product a weight per cu.ft of 163-167 lbs.

Continuous Test No. 2

Preliminary to starting this test the furnace was emptied of practically all of ^{the} preceding charge. The initial charge to the large end of the furnace was made up from the discharge remaining in the furnace from previous test namely about 280 lb plus 50 lb of coal and the initial charge to the hopper feed consisted of some 140 lb low metallic magnetics plus 90 pounds coal and 45 pounds coke.

When the temperature in the furnace had attained 930°C this temperature was maintained for two hours before the discharge pipe was opened. Conditions from beginning to end of the test were quite normal and no serious trouble was experienced, except in the discharge, which blocked occasionally due to lumps in the furnace. The approximate average temperature throughout the run was 1000-1038°C and the run lasted 104 hours. High metallization was obtained from start to finish as shown in the following table of the daily record.

Hrs.	Feed lbs.	Feed/hour lbs.	Disch. lbs.	Disch/hour lbs.	Mag. lbs.	Midd. lbs.	Non-Mag. lbs.	% Met. Fe		Oil consumed gals.
								Low	High	
24	2550	106.3	1699	70.8	1047.5	140	519	41.37	44.79	50
24	2400	100	1481	61.7	880.5	110	489	42.50	44.79	40
24	2650	110.4	1719.5	71.6	1071	88.5	560	41.25	43.95	50
24	2550	93.6	1556	64.8	960.5	60.5	535	41.46	43.02	40
8	350		549	68.6	332	34	84	40.94	42.09	20
104	10200	98.1	79 4.5	67.35	4291.5	433	2187			190

The total quantity of ore charged to the furnace was 5660 lbs. of coal 3015 lbs. and of coke 1520 lbs. Of the furnace discharge it will be noted that 61.3% is magnetic, 6.2% middling, and 31.5% tailing product. The middling product showed metallic iron content as follows:

First 24 hours	..	28.58	to	34.58	% metallic Fe
Second "	"	29.90		33.75	" "
Third "	"	26.25		27.18	" "
Fourth "	"	19.37		26.25	" "
Final 8	"	24.79			" "

The total middling product would average about 28% metallic iron and it is possible that by fine grinding and magnetic separation on a different type magnetic separator that a satisfactory recovery could be made of highly metallized concentrate. It might, however, be more economical and more convenient to pass this product with a coal mix back to the feed end of the furnace and treat with regular feed.

An examination of the tailing product which averages about 34% ash showed the following results:

Mesh	Weight per cent	Ash per cent	TiO ₂ Per cent	Fe per cent	Ash analysis	
					TiO ₂ %	Fe %
+14	52.54	26.40	1.51	3.59	5.71	10.11
-14+28	19.94	35.52	1.79	3.09	5.03	8.69
-28+48	14.34	48.80	3.26	4.85	6.68	9.93
-48+100	8.70	44.96	3.35	5.02	7.45	11.18
-100	4.46	47.70	3.39	5.82	7.11	12.20
Flue dust		70.35	17.40	22.86	24.73	32.48

The flue dust collected from the dust chamber amounted to about 3 per cent of the total feed charge.

Whether this tailing product can be advantageously utilized again has not been definitely determined. An attempt was made to use it mixed with low grade magnetic concentrates and middlings in the proportion of 100 parts magnetic, 60 tailing, and 20 of coke, without the addition of any fresh coal, and run it through the furnace in the usual manner. The result was not by any means encouraging as none of the magnetically separated discharge product analysed above 39.4% metallic iron and in comparison with the material used in the feed appeared to be changed but little. There is, however, a possibility of the tailing product being utilized after screening or other method of separation, in place of coke, to a limited extent, but time has not permitted securing any data along this line and the suggestion is therefore just a matter of personal opinion.

Referring back to the discharge system and the possible effect of oxidation of metallic iron occurring in the falling of the discharge product through air into the discharge bin, a sample of magnetic concentrate was examined as follows: A 2000 gram sample was screened on an 8 mesh screen, the products weighed and analysed for metallic iron and the - 8 mesh again screened through 48 and 65 mesh and the products analysed. A magnetic concentrate just below the desired grade in metallic iron content was used and the following table shows the results:

Mesh	Weight %	Metallic Fe %
Head		38.74
+8	20	41.10
-8	80	38.14
+48	93.2	38.96
-48+65	3.7	26.72
-65	3.1	23.35

The result here obtained would indicate that the finer particles are the more readily oxidized as would be expected.

Conclusions

The rotary kiln type of furnace seems to be quite satisfactory for this metallization process. An improvement in the discharge rate would tend toward better efficiency. This might be brought about in several ways. (1) by reducing the size of

coal and coke to the same size as the ore, (2) by increasing somewhat the large end diameter with a relative increase in the discharge pipe diameter. Finer grinding of the ore is not to be recommended in face of the results obtained and shown above. A good depth of bed charge must be maintained throughout, and the temperature must be held at not less than 990 C and not over 1050 C

Segregation occurs to some extent in the large diameter of the kiln, but this is not serious, its only effect apparently being in a varying discharge period weight. During operation it was observed that the coke had a tendency to ride on the surface of the charge. This it is believed is a highly satisfactory condition as it protects the ore bed from oxidation through air entering the furnace at the burner opening.

The degree of metallization obtained, namely 90-95% should be quite satisfactory for producing a low iron titanium dioxide concentrate. A metallized product analysing 43.35% metallic iron equivalent to 93.9% metallization gave on leaching with 10% sulphuric acid, a concentrate containing - titanium dioxide 75.89%, iron 6.27%, and carbon 0.80%.

The reducing agents used in these tests are perhaps more expensive than the process would economically warrant, but there is no reason to believe that less expensive fuels would be less satisfactory, provided that fuel of non-coking or very slightly coking type is used. It would be interesting to determine if anthracite fines could be utilized, without coke admixture, and if a low volatile matter coal could be used with equally successful results.

These tests were productive of nearly 4 tons of magnetic concentrate analysing over 40% metallic iron.