

**Bureau of Mines  
Report of Investigations 5170**



**ELECTRIC SMELTING OF ILMENITE CONCENTRATES  
FROM VALLEY COUNTY, IDAHO**

**BY L. H. BANNING, W. F. HERGERT, AND D. E. HALTER**

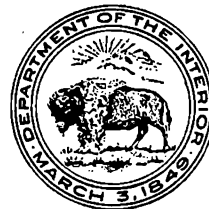
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**UNITED STATES DEPARTMENT OF THE INTERIOR  
Douglas McKay, Secretary  
BUREAU OF MINES  
J. J. Forbes, Director**

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Work on manuscript completed February 1955. The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is made: "Reprinted from Bureau of Mines Report of Investigations 5170."

**November 1955**

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by

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## CONTENTS

	<u>Page</u>
Summary .....	1
Introduction .....	1
Acknowledgments .....	2
Theoretical and practical considerations .....	2
Raw materials .....	4
Ilmenite concentrates .....	4
Reductants .....	5
Smelting equipment .....	5
Smelting campaigns .....	6
Tests in carbon-lined furnace .....	6
Tests in magnesite-lined furnace .....	11
Use of manganese ore in furnace charge .....	14
Discussion of smelting-test results .....	14
Effect of reductant on product characteristics and furnace operation .....	14
Recovery and grade of products .....	15
Electric power and electrode requirements .....	18
Conclusions .....	18

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## TABLES

	<u>Page</u>
1. Analyses of test lots of Idaho ilmenite concentrates .....	4
2. Summary operating data and test results in carbon-lined furnace .....	12
3. Summary operating data and test results in magnesite-lined furnace .....	13
4. Effect of manganese additions on Mn and Fe analysis of slag	14
5. Effect of amount and type of reductant on slag analyses ...	15
6. Typical analyses of various titanium raw materials .....	17
7. Spectrographic analyses of typical slag and pig iron products .....	17
8. Typical analyses of pig iron products, percent .....	17

## ILLUSTRATIONS

Fig.

1. Tapping viscous, high-titanium slag .....	7
2. Tapping pig iron .....	8
3. Pigging iron from ilmenite smelting .....	9
4. Segment of carbon lining from furnace showing erosion of key brick .....	10
5. High-titanium slag decrepitating on cooling .....	16

## SUMMARY

Recent dry-top electric smelting tests at the Northwest Electrodevelopment Experiment Station, Albany, Oreg., indicate that a high-titanium slag and a good grade of pig iron can be produced from alluvial Idaho ilmenite. A total of 120 tons of concentrates were smelted during this investigation, to yield an average of 0.5 ton of slag and 0.3 ton of pig iron per ton of ilmenite.

Power consumption averaged 1,400 kv.-hr. per ton of ilmenite, at a feed rate of 400 to 500 pounds per hour, when operating at an electrical energy input of 290 to 370 kilowatts. The optimum operating voltage appeared to be 110 volts, phase-to-phase. Graphite electrode consumption averaged 15.6 pounds per ton of ilmenite smelted. Magnesite furnace refractory withstood the corrosive action of the hot metal and slag better than carbon refractory.

During these tests, types and amounts of reductant required to reduce the iron from the ilmenite and produce a slag having the highest possible titanium content were studied. Fluidity of slag determined the upper limit of reduction, as titanium slags containing less than 10 percent Fe-plus-Mn were very viscous at usual operating temperatures. Hotted fuel and coke were the reductants used. Slags containing between 67 and 86 percent  $TiO_2$  were produced. The  $TiO_2$  content of the majority of the slags varied between 80 and 84 percent and averaged about 4.5 percent Mn and 0.5 percent  $Cb_2O_5$ . The Fe content varied inversely to the  $TiO_2$  content, usually between 7 and 9 percent. The amount of Ti reduced to metal was insignificant.

During a 40-hour period, high-grade manganese ore was added to the furnace charge to determine whether the manganese would replace the iron in the slag. The most logical use of the slag product is for the production of titanium tetrachloride, which is used by the titanium metal industry. Iron chloride is a waste product of such an operation and manganese chloride might be a valuable byproduct. The results of the test were encouraging; during one 8-hour shift the average iron content of the slag was only 2.1 percent.

The presence of  $Cb_2O_5$  in the slag, approximately 0.53 percent, is also of much interest, as there is a possibility of recovering columbium as a valuable coproduct in a slag-chlorination operation.

## INTRODUCTION

Although titanium is one of the most abundant metals in the earth's crust, minerals rich in this element are few; only ilmenite ( $FeTiO_3$ ) and rutile ( $TiO_2$ ) are of commercial importance. Titanium-bearing minerals such as anatase, leucoxene, brookite, and others are associated with ilmenite and rutile and usually comprise part of the ilmenite and rutile ore marketed. In some localities, ilmenite has been altered by weathering and contains  $TiO_2$  above the theoretical content. Ilmenite, the most abundant of the commercially available titanium minerals, is recovered from both lode and placer deposits and is chiefly consumed in the titanium pigment

industry. Rutile is the highest grade titanium mineral commercially available, but it is also the least abundant and the most costly per unit of titanium. It is used mainly for the production of titanium by the Kroll process and in coatings for welding rods.<sup>2/</sup> Titanium slag now produced from ilmenite-hematite ore is also used mainly in the titanium pigment industry.<sup>3/</sup>

The large expansion of the domestic titanium metal industry proposed within the next 10 years has prompted much research in all phases of the industry, from investigating suitable raw materials to fabricating the final product. In addition to rutile, it will probably be necessary to use ilmenite, and possibly titaniferous magnetites, to provide enough raw materials for the future production of titanium metal.

Ilmenite produced as a byproduct of the monazite sand-dredging operations in Valley County, Idaho, is particularly desirable for processing into a slag product suitable for metal production because of its low content of such chloride consumers as MgO, CaO, SiO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub>.<sup>4/</sup> This ilmenite is being produced and stockpiled continuously. The long distance from ilmenite-consuming areas and its comparatively low TiO<sub>2</sub> content has made it impractical to market.

The Bureau of Mines began research at the Northwest Electrodevelopment Experiment Station, Albany, Oreg., early in 1954 to develop a smelting process for utilizing this ilmenite. The principal objective of the research was to develop a satisfactory electric smelting process for producing a high-titanium slag and pig iron. Development of a technical and economically feasible process for utilizing this ilmenite would not only encourage monazite production, which is a marginal operation now, but would also provide material for production of titanium dioxide and metal.

#### ACKNOWLEDGMENTS

The cooperation of officials of Baumhoff-Marshall, Inc., and the Idaho-Canadian Dredging Co. for furnishing the ilmenite used in these tests is gratefully acknowledged.

#### THEORETICAL AND PRACTICAL CONSIDERATIONS

Previous electric smelting tests on ilmenite had been made at the Bureau of Mines laboratory at Boulder City, Nev.,<sup>5/</sup> using 500 and 2,500 pounds of ilmenite per batch. Some fluxes were used in the furnace charges to fluidize the slag, and the final product contained up to 70 percent TiO<sub>2</sub>. The Quebec Iron and Titanium Corp. produces a titanium slag from Allard Lake, Quebec, ilmenite containing about 72 percent TiO<sub>2</sub>.<sup>6/</sup> No flux is used in the furnace charge but the ilmenite contains appreciable amounts of the usual fluxing constituents (chloride consumers). This

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<sup>2/</sup> Gillson, J. L. Titanium: AIME, Industrial Minerals and Rocks, 2d ed., New York, N. Y., 1949, pp. 1042-1070.

<sup>3/</sup> Knoerr, A. W. World's Major Titanium Mine and Smelter Swing into Fullscale Production: Eng. Min. Jour., vol. 153, No. 3, March 1952, pp. 72-79.

<sup>4/</sup> Opie, W. R. Titanium Extraction by Chloride Process Presents a Variety of Problems: Jour. Metals, vol. 6, No. 7, July 1954, pp. 807-810.

<sup>5/</sup> Stoddard, C. K., Cole, S. S., Eck, L. T., and Davis, C. W. Pilot-Plant Smelting of Ilmenite in the Electric Furnace: Bureau of Mines Rept. of Investigations 4750, December 1950, 15 pp.

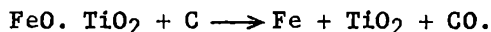
<sup>6/</sup> See work cited in footnote 3.

slag is used mainly in the pigment industry. The primary objective of the tests on Idaho ilmenites was to produce a slag containing enough  $\text{TiO}_2$  (80 to 85 percent) to be useful for the production of metal by the Kroll process.

It is desirable to produce a high titanium slag without adding flux when the slag is to be used for either the production of metal or pigment. According to Grieve and White<sup>7/</sup> the melting point decreases as the  $\text{FeO}$  is reduced from 47 to 32 percent. At 32 percent  $\text{FeO}$  a low-melting eutectic occurs. As the iron content diminishes below that of the eutectic, the melting point increases at a constant rate. However, if the degree of reduction is great enough, some  $\text{TiO}_2$  is reduced to  $\text{Ti}_2\text{O}_3$  before all the  $\text{FeO}$  is reduced from the slag. The melting point of slag containing appreciable amounts of  $\text{Ti}_2\text{O}_3$  is very high, and the maximum titanium content obtainable in the slag is limited by the temperature at which the slag can be tapped from the furnace.

Previous smelting research at the Northwest Electrodevelopment Experiment Station has shown that the temperature maintained in an electric smelting furnace depends mainly upon the type of reductant used and the electrical energy input to the furnace. The value of a bulky reductant, such as wood chips in the form of hogged wood, for controlling smelting temperatures has been described by the Bureau.<sup>8 9/</sup> The reductant may be a 100-percent wood chips, which gives the lowest bulk density charge and the highest smelting temperature, or mixtures of wood chips and denser forms of carbon such as coke. Large proportions of dense forms of carbon in the charge tend to reduce the smelting temperature. To operate an electric arc furnace at the highest attainable energy input, proper electrical conditions must be maintained. Methods used for selecting the proper electrical conditions for a particular smelting operation also have been described.<sup>10/</sup> Before the tests were started, it was recognized that it would be necessary to use a comparative low voltage because of the high electrical conductivity of titanium slags. The effect of  $\text{FeO}$  content on the electrical conductivity of titanium slags has been studied by Wyatt.<sup>11/</sup>

Thermodynamic studies by Bureau investigators have shown that iron in ilmenite can be effectively reduced by carbon but not by carbon monoxide.<sup>12/</sup> In calculating the amount of carbon needed for the smelting tests, the reducing effect of carbon alone was considered; the calculated carbon requirement was based on the following reaction:



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- <sup>7/</sup> Hall, F. P., and Insley, H. Phase Diagrams for Ceramists: Am. Ceram. Soc. Jour., vol. 30, No. 11, pt. 2, Nov. 1, 1947, p. 28.
- <sup>8/</sup> Rasmussen, R. T. C. Electric Smelting at Bureau of Mines Seeks Utilization of Northwest Ores: Jour. Metals, vol. 4, December 1952, pp. 1273-1279.
- <sup>9/</sup> Cremer, Herbert. Continuous Electric Smelting of Low-Grade Nickel Ores: Bureau of Mines Rept. of Investigations 5021, 1954, 36 pp.
- <sup>10/</sup> Banning, L. H. The Role of the Electric Arc Furnace in Utilizing Some Strategic Off-Grade Ores: Jour. Electrochem. Soc., vol. 101, No. 12, December 1954, pp. 613-621.
- <sup>11/</sup> Wyatt, J. L. The Electrical Resistivity of Titanium Slags: Trans. AIME, vol. 188, August 1950, pp. 989-994.
- <sup>12/</sup> Shomate, C. H., Naylor, B. F., and Boericke, F. S. Thermodynamic Properties of Ilmenite and Selective Reduction of Iron in Ilmenite: Bureau of Mines Rept. of Investigations 3864, May 1946, 19 pp.

Freundlich's studies indicated that from 13 to 16 grams of charcoal were required per 100 grams of ilmenite reduced.<sup>13/</sup> His investigation also indicated that if too much carbon was used in the charge, at certain specified smelting temperatures, the iron did not readily separate from the slag. The Bureau planned to use hogged wood as the major source of carbon; 100 pounds of run-of-mill hogged wood or wood chips per 100 pounds of ilmenite is approximately equivalent in carbon content to the charcoal used in the ilmenite-reduction experiments by Freundlich.

## RAW MATERIALS

### Ilmenite Concentrates

The ilmenite for this series of tests consisted of sand concentrates produced as a byproduct of existing monazite sand-dredging operations. The concentrates were furnished jointly and on an equal basis by the Idaho-Canadian Dredging Co. and Baumhoff-Marshall, Inc., both with headquarters in Boise, Idaho.

Since these concentrates were from the same locality in Valley County, Idaho, the chemical and physical characteristics were quite similar. Amounts, source, and analyses of the six individual shipments are tabulated in table 1.

TABLE 1. - Analyses of lots of Idaho ilmenite concentrates

Lot No. ....	1	2	3	4	5	6
Source .....	1/I-C	2/B-M	I-C	I-C	B-M	B-M
Pounds .....	42,730	42,590	42,690	39,290	36,680	44,290
Analysis, percent:						
Fe .....	34.8	34.0	35.1	34.9	35.0	34.2
TiO <sub>2</sub> .....	42.4	41.0	41.0	42.2	44.8	39.2
CaO .....	0.30	0.50	-	-	-	-
MgO .....	0.10	0.10	0.10	0.10	-	-
Al <sub>2</sub> O <sub>3</sub> .....	0.70	0.20	1.60	1.20	0.82	1.90
SiO <sub>2</sub> .....	1.40	0.56	1.00	0.50	1.60	1.60
Mn .....	2.86	3.10	3.09	3.20	2.92	2.86
Cb <sub>2</sub> O <sub>5</sub> .....	0.24	0.27	0.25	0.26	0.32	0.30

1/ Idaho-Canadian Dredging Co.

2/ Baumhoff-Marshall, Inc.

The manganese and columbium contents are of special interest. According to currently available information, these particular concentrates contain higher percentages of these two elements than similar ilmenite from alluvial deposits in other parts of the world.

In addition to the above elements, spectrographic analysis shows the following to exist in amounts of less than 0.1 percent; As, Cu, Zn, Cr, V, and B.

Petrographic examination of the different lots indicate that 67 to 81 percent of the mineral exists in the form of ilmenite with intergrown magnetite, 16 to 30 percent in the form of ilmenite with small amounts of leucoxene, and very small amounts as magnetite and garnet. These combinations of minerals account for 99 percent of the total. Screen analyses were made on the concentrate shipments as

<sup>13/</sup> Freundlich, W. Quantitative Study of Extraction of Titanium Dioxide From Ilmenite by Smelting, Part II: Bull. soc. chim., vol. 19, No. 5, 1952, pp. 496-501. (Harry Brutcher Translation No. 3120, 1953.)



received. An average of approximately 75 percent reported in the size range of minus-28- plus-48-mesh. Only a small percentage was plus-28-mesh. The remarkable uniformity of the grain size can be attributed to the alluvial source.

#### Reductants

Wood chips, hogged wood, and metallurgical coke were used as reductants. Wood chips and hogged wood not only served as reductants, but were also used as bulk control for smelting. Both wood waste products were obtained locally.

The wood chips are a quite uniform product made from scrap plywood veneer in a machine known as a chipper. The product is a chip approximately 1/8 inch thick and 5/8 inch square free of splinters and sawdust. Hogged wood, a variable mixture of splinters, chips, and sawdust, is produced by running sawmill woodwaste scraps through a machine known as a hog. Only small amounts of hogged wood were used during this test, the major proportion of reductant being wood chips. The bulky reductant was sampled for moisture and fixed carbon systematically throughout the tests. Shift samples and 24-hour composite samples were taken for moisture and fixed-carbon determinations. Check samples for moisture control were taken at regular intervals and dried in a commercial Moisture Teller. The series of samples taken on a shift basis averaged 40.6 percent moisture on the as-received basis, and contained 23.5 percent fixed carbon on a dry basis.

In these tests relatively small amounts of metallurgical coke were used. This coke contained from 79 to 81 percent fixed carbon on a dry basis.

#### SMELTING EQUIPMENT

The furnace used in these tests was a 3-phase, cylindrical, stationary, pit-type electric arc furnace. It has a shell 50 inches in diameter and 55 inches high. Two types of lining were used in the furnace, a carbon lining and the other a magnesite lining. For the carbon lining, standard 9-inch carbon key brick backed by 2-1/2-inch-thick standard magnesite brick were used. This arrangement formed a crucible 27 inches in diameter. Two tap holes were installed, one 7-1/2 inches above the other. The hearth of the furnace consisted of several layers of carbon brick covered with enough carbon ramming material to form a dish-shaped bottom.

For the magnesite lining, only standard 9-inch key brick were used, giving a crucible 32 inches in diameter and 46 inches in depth. One tap hole was installed 5 inches above the other in this larger crucible. The hearth of the magnesite furnace was built up in a similar manner to that of the carbon hearth, except that magnesite brick and a commercial periclase ramming material were used. In both furnaces, the lower tap hole was installed level with the hearth so that the furnace could be completely drained when tapping from this hole.

The furnace uses either 3- or 4-inch graphite electrodes. The electrode clamp arm assemblies are cable-suspended, with provisions for changing the electrode spacing. The electrodes are placed at the corners of an equilateral triangle. Electrode current was automatically controlled by a balanced beam-type regulator.

The furnace is connected to a 1,000 kv.-a. Westinghouse transformer, which has 6 voltage taps. The secondary buses of the transformer may be connected in either series or parallel. The open circuit, phase-to-phase voltage ranges from 38 to 106 volts on the parallel and from 80 to 220 volts on the series connection.

Necessary auxiliary equipment was available for conducting the tests efficiently, including a 6-cubic-foot concrete mixer for mixing the charge, a hoist for lifting the charge bucket to the charge deck, conical cast iron slag thimbles, pig molds, and refractory-lined ladles.

### SMELTING CAMPAIGNS

Before feeding the ilmenite charges, the furnace was preheated by arcing on a bed of coke on the hearth for several hours. The coke was then removed from the furnace and pig iron was melted on the hearth to distribute the heat evenly throughout the refractories. After tapping the pig iron from the furnace the test was started by charging ilmenite concentrates mixed with wood chips. When a normal operation was attained, 2 to 3 feet of loose charge covered the molten material in the furnace.

Usually, slag was tapped from the upper tap hole into a conical cast iron slag thimble at approximately 1-1/2-hour intervals. When metal appeared in the slag taps, at intervals of about 6 hours, the lower tap hole was opened and the pig iron drained into a refractory-lined ladle. During one period in which the charge was particularly high in carbon, slag and pig iron were tapped simultaneously from the lower tap hole. When using this procedure it was necessary to tap both slag and metal into a refractory-lined ladle and decant the slag into cast iron thimbles. This proved to be a difficult operation because the high-melting point slag would rapidly consolidate on the ladle lining. Furnace operations were also more difficult, and indications were that the slag formed false bottoms in the furnace, which were difficult to penetrate when tapping. Consequently, the original tapping procedure was reverted to for the remainder of the smelting campaign.

#### Tests in Carbon-Lined Furnace

The first two of the six lots of ilmenite were smelted in the carbon-lined furnace. The preliminary test was divided into periods A, B, C, and D. Only wood chips were used as reductant in an attempt to attain the highest possible smelting temperatures. During the first 3 periods, the weight of the wood chips, per 100 pounds of ilmenite, was increased from 75 to 100, and then to 115 pounds. In period A, the slag-tapping temperature and the  $\text{TiO}_2$  content of the slag samples were considerably lower than anticipated. The wood chip proportion was increased in period B. This change resulted in production of a lower iron content slag, but there was only a slight increase in the smelting temperature. To investigate this trend further more reductant was added to the charge during period C. A very viscous slag was produced, which froze to the furnace walls and caused furnace-operating difficulties. Figure 1 illustrates the tapping of a viscous slag. This forced a cutback in reductant in period D to return the furnace to an operable condition.

There was evidence during this preliminary test that the carbon lining was being dissolved by the pig iron. Figures 2 and 3 show the tapping and pouring of pig iron into molds. The accompanying shower of sparks appears to indicate the metal's high carbon content. After 93 hours of continuous operation, iron began leaking through the furnace bottom and the test was terminated. Before smelting was resumed the furnace lining was disassembled and carefully examined. Figure 4 is a view of a segment of the lining showing how the pig iron had attacked the carbon brick.

To establish definitely whether carbon refractory is unsuitable for smelting ilmenite, the furnace was relined with carbon brick for the second series of

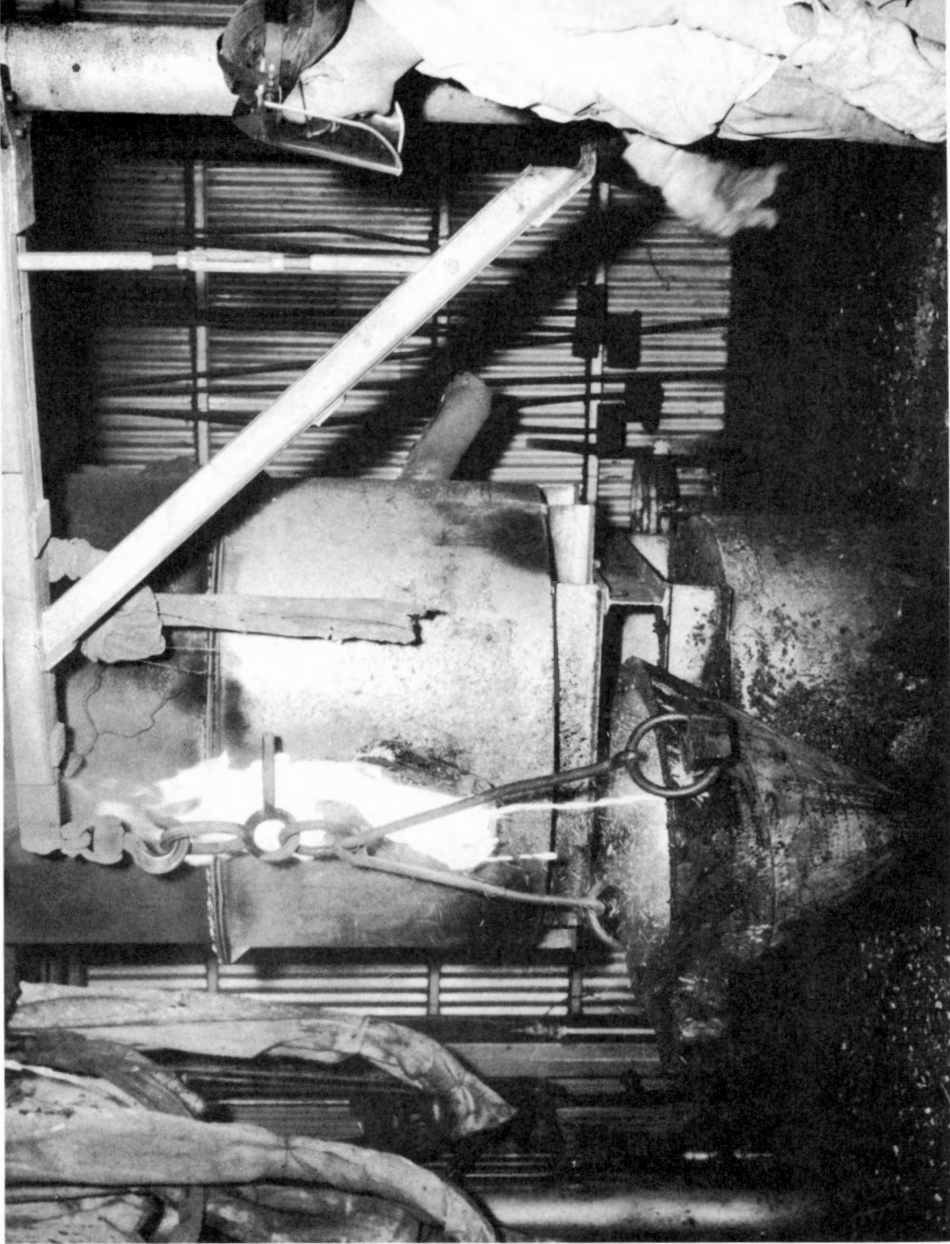


Figure 1. - Tapping viscous, high-titanium slag.

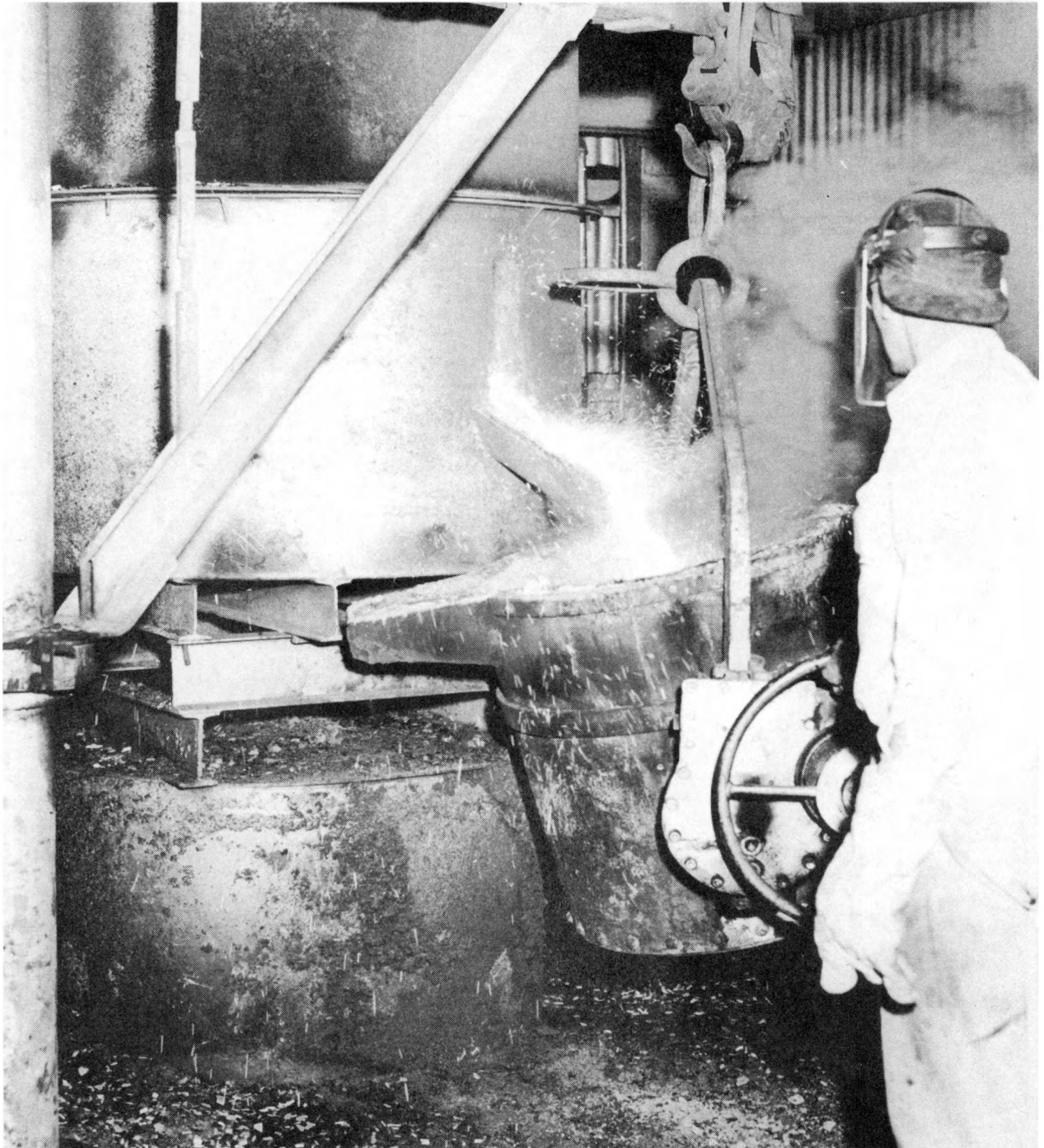


Figure 2. - Tapping pig iron.

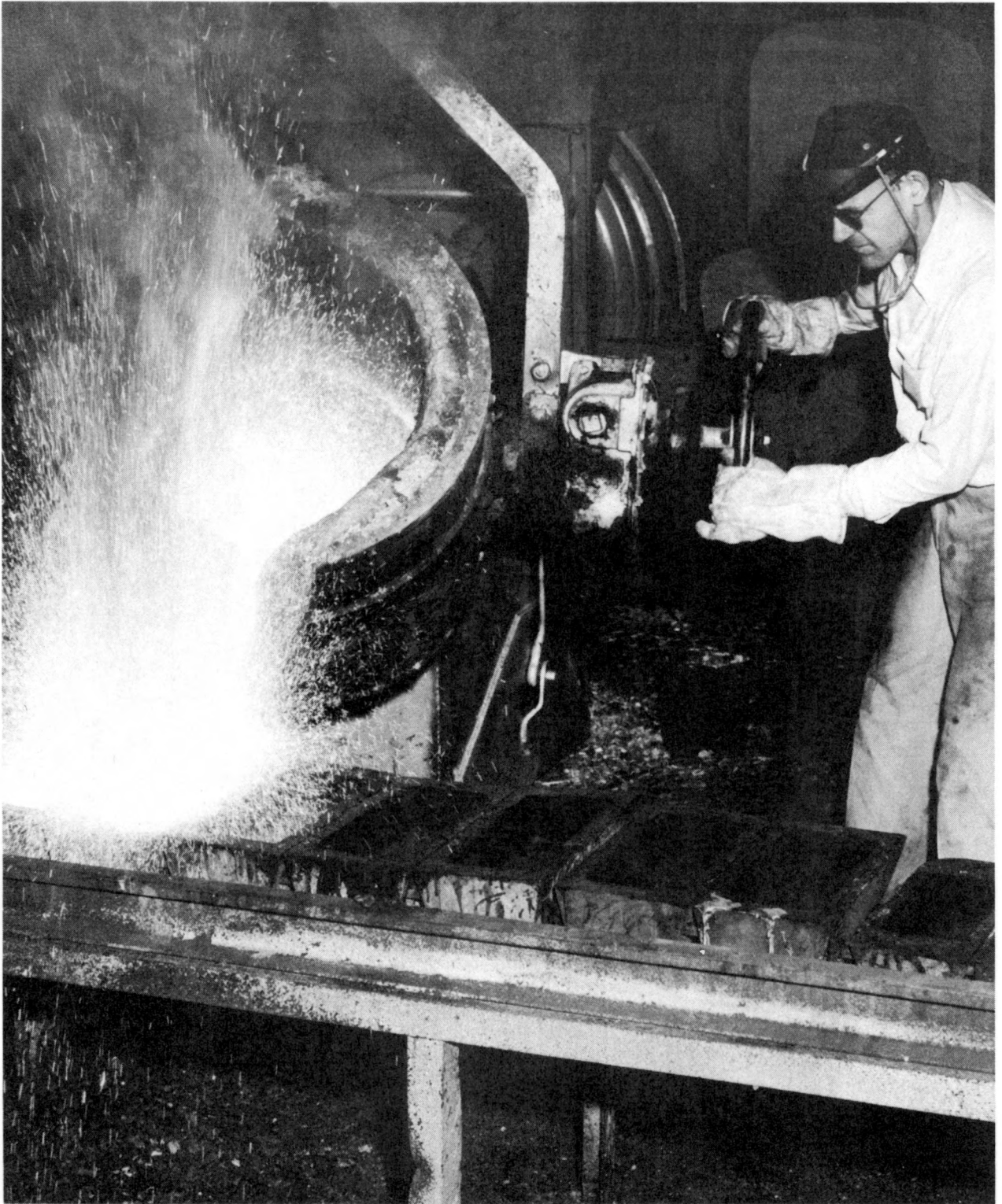


Figure 3. - Pigging iron from ilmenite smelting.





Figure 4. - Segment of carbon lining from furnace, showing erosion of key brick.

smelting tests. The most promising furnace-operating conditions developed during the first test were selected. The test was divided into periods A and B; during period A, only hogged wood was used for the reductant; in period B, a small amount of coke was added to the charge and the electrical energy input was increased about 15 percent. During the fourth day of operation, pig iron again leaked through the furnace bottom. Operations were interrupted long enough to permit the iron to drain through the hole, which then filled with solidified slag. There was no recurrence of a metal break-through during the 20 hours required to finish the smelting tests.

A summary of the data for tests in the carbon-lined furnace is shown in table 2.

### Tests in Magnesite-Lined Furnace

As metal breakthroughs had occurred in both tests in the carbon-lined furnace, a magnesite lining was used for the remaining tests. Ilmenite lots 3, 4, and 6 were smelted in a continuous 268-hour operation. After a week-end shut down, lot 5 was smelted in the same lining. Operating data and results of the smelting tests are presented in table 3.

The smelting tests on all the lots except lot 6 were divided into two periods. To insure a fluid, easy tapping slag, smelting was started on lot 3 using wood chips only and in smaller amounts than that required to produce a high titania slag. After furnace thermal equilibrium was attained, an additional 25 pounds of wood chips per 100-pound ilmenite charge were used. During period B, coke was gradually added to the charge until a maximum of 2.5 pounds per 100-pound charge was being used.

After lot 3 was consumed, smelting of lot 4 continued under essentially the same conditions. During the latter part of period A, the slag became so viscous that it was necessary to decrease the reductant, and coke was eliminated from the charge. The composite 8-hour slag sample for the last shift in which coke was used analyzed 84 percent  $\text{TiO}_2$ . When furnace operations had returned to normal the  $\text{TiO}_2$  content had decreased to 78 percent. In the latter part of period B, coke was again added to the charge, and the  $\text{TiO}_2$  content of the slag increased to 83 percent.

Smelting was continued without interruption on lot 6 ilmenite. The effect of substituting coke for wood chips was investigated further; reductant proportions ranged from 62 pounds of wood chips and 5 pounds of coke to 100 pounds of wood chips and no coke, per 100-pound ilmenite charge. A lesser amount of total carbon was used in this test than in any of the others, and the average  $\text{TiO}_2$  content of the slag was less. Several changes in charge proportions caused some furnace operating difficulties, which at times produced a low-grade slag product.

After smelting lots 3, 4 and 6 in a continuous operation, the furnace was shut down for the week end. During the shutdown the magnesite brick spalled considerably. Consequently, when smelting was resumed on lot 5 ilmenite, unforeseen difficulty was experienced with a gummy, sticky slag because pieces of magnesite were melted in the slag. Once they were consumed, furnace operation returned to normal.

TABLE 2. - Summary operating data and test results in carbon-lined furnace

Lot No. ....	Lot 1					Lot 2		
	A	B	C	D	Whole test	A	B	Whole test
Period .....	7.13	16.13	7.80	61.57	92.63	41.75	51.67	93.42
Duration of test ..... hr.								
Charge proportions per 100 lb. ilmenite:								
Wood chips <sup>1/</sup> .....	75	100	115	100	-	100	100	-
Coke <sup>2/</sup> .....	-	-	-	-	-	-	1	-
Feed rate..... lb. ilmenite per hour	448.8	372	410.3	422.3	414.5	421	476	451.6
Total materials charged, lb.:								
Ilmenite .....	3,200	6,000	3,200	26,000	38,400	17,585	24,600	42,185
Wood chips .....	2,400	6,000	3,680	26,000	38,080	17,585	24,600	42,185
Coke .....	-	-	-	-	-	-	246	246
Ratio of stoichiometric carbon <sup>3/</sup> .....	143	191	201	191	189	186	197	193
Electrical conditions:								
Voltage ..... phase-to-phase	115	110	118	110	114	110	108	108
Amperes .....	1,480	1,540	1,500	1,585	1,540	1,750	1,768	1,759
Kilowatt input .....average	251	245	299	287	279	287	340	317
Electric power consumption, kw.-hr.:								
Total .....	1,790	3,950	2,330	17,730	25,800	11,970	17,610	29,580
Per ton ilmenite .....	1,119	1,317	1,456	1,364	1,344	1,361	1,432	1,402
Per ton titanium slag .....	2,882	2,228	2,342	2,508	2,467	2,356	2,581	2,484
Per ton pig iron .....	4,207	3,976	5,884	6,218	5,529	5,139	5,345	5,260
Graphite electrode consumption, lb.:								
Total .....	-	-	-	-	271.2	-	-	308.5
Per ton ilmenite .....	-	-	-	-	14.1	-	-	14.6
Per ton titanium slag .....	-	-	-	-	25.9	-	-	25.9
Per ton pig iron .....	-	-	-	-	58.1	-	-	54.9
Products, lb.:								
Titanium slag .....	1,242	3,545	1,990	14,140	20,917	10,163	13,648	23,811
Pig iron .....	851	1,987	792	5,703	9,333	4,658	6,589	11,247
Titanium slag analysis, percent:								
TiO <sub>2</sub> .....	66.9	73.1	82.5	77.5	76.6	80.6	82.7	81.8
Fe .....	13.8	11.5	7.2	9.9	10.2	9.8	8.0	8.8
Mn .....	4.49	4.49	4.49	4.44	4.46	4.42	4.57	4.51
Pig iron analysis, percent:								
Fe .....	93.2	93.3	93.7	94.8	94.3	95.2	95.4	95.3
C .....	3.37	4.46	4.97	3.82	4.04	4.48	3.96	4.17
Ti .....	0.23	0.25	1.20	1.14	0.88	0.08	0.08	0.08
Mn .....	0.35	0.47	0.19	0.19	0.26	0.12	0.18	0.15
Recovery, percent:								
TiO <sub>2</sub> in slag .....	-	-	-	-	4/99.8	-	-	5/108.9
Mn in slag .....	-	-	-	-	79.0	-	-	89.0
Fe in metal .....	-	-	-	-	67.4	-	-	75.5
Accounted for in products:								
TiO <sub>2</sub> .....	-	-	-	-	100.3	-	-	108.9
Fe .....	-	-	-	-	83.8	-	-	89.9
Mn .....	-	-	-	-	81.0	-	-	90.4
Average slag tapping temperature ..... °C.	1,560	1,579	1,615	1,626	1,609	1,598	1,605	1,602

<sup>1/</sup> Wood chips, 23.5 percent F. C. dry basis, 40.6 percent moisture as received.

<sup>2/</sup> Coke 79.0 percent F. C. dry basis, 4 percent moisture as received.

<sup>3/</sup> Stoichiometric carbon for complete reduction of iron only.

<sup>4/</sup> After magnetic separation of iron prills from slag.

<sup>5/</sup> High TiO<sub>2</sub> recovery and low iron recovery due to iron entrapped in viscous slag.



TABLE 3. - Summary operating data and test results in magnesite-lined furnace

Lot No. ....	Lot 3			Lot 4			Lot 6	Lot 5		
	A	B	Whole test	A	B	Whole test	A	A	B	Whole test
Period .....	46.16	70.42	116.58	49.83	45.67	95.5	66.08	60.08	35.00	95.08
Duration of test ..... hr.										
Charge proportion per 100 lb. ilmenite:										
Wood chips <sup>1/</sup> .....	75 to 100	100	-	100	72	-	62 to 100	75 to 90	75 to 90	-
Coke <sup>2/</sup> .....	-	0 to 2½	-	0 to 1	2½	-	5 to 0	1½ to 0	2½ to 1½	-
MnO .....	-	-	-	-	-	-	-	-	3½ to 7	-
Feed rate...lb. ilmenite per hr.	364	354	358	369	395	381	439	498	442	477
Total materials charged, lb.:										
Ilmenite .....	16,800	24,917	41,717	18,400	18,025	36,425	29,000	29,900	15,470	45,370
Wood chips .....	15,035	24,917	39,952	18,400	13,306	31,706	20,516	24,810	12,966	37,776
Coke .....	-	274	274	92	448	540	1,112	174	279.4	453.4
MnO <sub>2</sub> .....	-	-	-	-	-	-	-	-	577.5	577.5
Ratio of stoichiometric C <sub>3</sub> /....	165.5	196	184	191	162	177	140.7	161	173.7	165
Electrical conditions:										
Voltage ..... phase-to-phase	81	90	87	94	97	96	97	102	96	100
Amperes .....	1,520	1,864	1,734	1,913	1,947	1,929	2,010	2,132	2,490	2,258
Kilowatt input.....average	249	273	263	269	262	266	290	321	316	320
Electric power consumption, kw.-hr.:										
Total .....	11,490	19,220	30,710	13,410	11,960	25,370	19,180	19,310	11,070	30,380
Per ton ilmenite .....	1,368	1,543	1,472	1,458	1,327	1,393	1,323	1,292	1,431	1,339
Per ton titanium slag .....	2,930	2,814	2,856	2,758	2,390	2,571	2,688	2,345	2,776	2,485
Per ton pig iron .....	4,770	5,698	5,311	4,784	4,924	4,849	4,830	4,557	4,193	4,417
Graphite electrode consumption, lb.:										
Total .....	-	-	473.5	-	-	324	92	-	-	417.5
Per ton ilmenite .....	-	-	22.7	-	-	17.8	6.3	-	-	18.4
Per ton titanium slag .....	-	-	44	-	-	32.8	12.9	-	-	34
Per ton pig iron .....	-	-	82	-	-	61.9	23.2	-	-	60.7
Products, lb.:										
Titanium slag .....	7,842	13,661	21,503	9,724	10,008	19,732	14,272	16,470	7,976	24,446
Pig iron .....	4,818	6,746	11,564	5,606	4,858	10,464	7,942	8,475	5,280	13,755
Titanium slag analysis, percent:										
TiO <sub>2</sub> .....	74.8	80.5	78.4	82.9	80.9	81.9	78.8	78.8	81.8	79.7
Fe .....	10.9	6.7	8.2	6.6	7.4	7.0	9.0	9.2	3.0	7.2
Mn .....	4.10	3.99	4.03	4.54	4.63	4.59	4.59	4.54	7.06	5.33
Cb <sub>2</sub> O <sub>5</sub> .....	0.54	0.52	0.53	0.53	0.55	0.54	0.56	0.50	0.22	0.41
Pig iron analysis, percent:										
Fe .....	96.3	96.7	96.5	96.6	96.6	96.6	96.9	96.9	96.3	95.7
C .....	3.12	3.00	3.05	3.17	3.05	3.11	2.80	2.69	2.48	2.59
Ti .....	0.06	0.05	0.05	0.05	0.10	0.07	0.07	0.06	0.11	0.08
Mn .....	0.44	0.23	0.32	0.26	0.09	0.18	0.14	0.26	0.78	0.46
Recovery, percent:										
TiO <sub>2</sub> in slag <sup>4/</sup> .....	-	-	95.8	-	-	99.0	95.5	-	-	106.7
Mn in slag .....	-	-	70.1	-	-	77.6	79.0	-	-	80.2
Cb <sub>2</sub> O <sub>5</sub> in slag .....	-	-	109.2	-	-	112.5	91.7	-	-	70.7
Fe in metal .....	-	-	76.2	-	-	79.5	77.6	-	-	84.4
Accounted for in products:										
TiO <sub>2</sub> .....	-	-	95.8	-	-	99.2	95.6	-	-	106.8
Fe .....	-	-	88.3	-	-	90.4	90.5	-	-	95.6
Mn .....	-	-	75.7	-	-	79.3	80.3	-	-	84.1
Cb <sub>2</sub> O <sub>5</sub> .....	-	-	115.4	-	-	118.7	98.1	-	-	89.9
Average slag tapping temperature ..... °C.	1,544	1,625	1,590	1,633	1,625	1,630	1,605	1,600	1,579	1,592

1/ Wood chips, 23.5 percent F.C. dry basis, 40.6 percent moisture as received.

2/ Coke, 79 percent F.C. dry basis, 4 percent moisture as received.

3/ Stoichiometric carbon for reduction of iron only. 4/ After magnetic separation of iron prills from slag.

### Use of Manganese Ore in Furnace Charge

When smelting lot 5 ilmenite a small amount of manganese ore was added to the charge during period B to determine whether manganese would displace iron from the slag, as manganese chloride may be a valuable byproduct of a titanium chlorinating operation when using these slags. At first, a stoichiometric amount of manganese was added to displace all of the iron in the slag. Under these conditions a considerable amount of manganese appeared to volatilize. When manganese additions were cut in half smelting operations returned to normal. The  $TiO_2$  content of the shift slag samples analyzed as high as 86 percent  $TiO_2$  and 7.1 percent Mn and as low as 2.4 percent Fe. Normally the slag contained about 4.5 percent Mn and 6 to 9 percent Fe. The Fe and Mn analysis of the slag taps during this period are shown in table 4.

TABLE 4. - Effect of manganese additions on Mn and Fe analysis of slag

Tap No.	Fe	Mn	Tap No.	Fe	Mn
50-52 .....	7.6	<u>1/</u> 4.64	69 .....	3.9	7.27
53-55 .....	6.2	<u>1/</u> 4.60	70 .....	4.1	7.53
57 .....	4.8	<u>2/</u> 5.35	71 .....	4.0	7.52
58 .....	3.0	8.45	73 .....	4.3	7.27
59 .....	4.3	6.88	74 .....	3.52	<u>3/</u> 6.96
61 .....	3.5	5.72	75 .....	4.56	6.04
62 .....	2.6	7.18	77 .....	3.60	6.96
63 .....	3.2	8.08	78 .....	4.04	6.86
64 .....	5.3	7.91	79 .....	4.12	7.30
66 .....	5.7	6.62	81 .....	4.08	6.83
67 .....	5.3	6.97	82 .....	4.00	6.19

1/ Before manganese additions.

2/ Started manganese additions.

3/ Stopped manganese additions.

### DISCUSSION OF SMELTING TEST RESULTS

#### Effect of Reductant on Product Characteristics and Furnace Operation

The ratio of total carbon to the stoichiometric proportion and the ratio of coke to wood chips were varied in the furnace charge during these investigations. Changing either of these ratios had an effect on both product characteristics and furnace operation. Examination of test results indicate the following trends when the total amount of reductant was increased:

1. An increase in the  $TiO_2$  content of the slag.
2. An increase in the carbon content of the pig iron.
3. An increase in power consumption per ton of slag produced.
4. A decrease in the iron content of the slag.
5. An increase in the smelting temperature.
6. The production of a more viscous slag, which increased tapping difficulties.

Petrographic examination of the high-titanium slag product revealed that it was a glassy, poorly crystallized material containing essentially anatase and rutile. Relatively small amounts of brookite and metallic iron were also identified. The melting point of ilmenite ( $\text{TiO}_2 \cdot \text{FeO}$ ) is low compared to that of  $\text{TiO}_2$ ,  $1,367^\circ \text{C}$ . and  $1,640^\circ \text{C}$ ., respectively. Although petrographic examination failed to reveal the presence of lower oxides of titanium such as  $\text{Ti}_2\text{O}_3$ , gain in weight on ignition in air indicated their presence. The melting point of  $\text{Ti}_2\text{O}_3$  has been reported to be  $2,130^\circ \text{C}$ . The ilmenite smelted contained low percentages of slag-forming constituents, such as  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ ; therefore the melting point of the slag depended mainly upon its Fe and Mn content, and on the presence of lower oxides of titanium. If too much reductant was added to the furnace charge, the slag became too viscous to be completely tapped from the furnace, and would consolidate on the furnace walls. To clear the accumulated slag from the furnace, it was necessary to reduce the amount of reductant in the charges. Consequently, the  $\text{TiO}_2$  content of the slag dropped when reestablishing normal furnace operation.

In some of the periods of the tests only wood chips were used for the reductant; in others both wood chips and coke were used. One of the reasons for using wood chips was to provide a high smelting temperature. However, test results indicated that increasing the weight of the wood chips to over 100 pounds per charge was not effective in increasing the smelting temperature. Table 5 is an arrangement of selected data from various shifts showing various ratios of total carbon to the stoichiometric proportion, the percentage of the carbon as coke, and the analyses of slag products. The use of some coke in the furnace charge appeared to be beneficial in producing slags of higher  $\text{TiO}_2$  content with an equivalent amount of total carbon and in reducing electrode consumption.

TABLE 5. - Effect of amount and type of reductant on slag analyses

Ratio of total C to stoich. C.	Percent of C as coke	Analysis, percent		Hours at condition
		$\text{TiO}_2$	Fe plus Mn	
1.56	None	73.9	16.6	32
1.68	38.6	77.9	13.4	56
1.74	16.7	81.1	11.7	40
1.80	8.9	81.0	11.3	40
1.86	None	77.3	14.3	56
2.10	do.	80.5	11.6	56
2.10	14.3	82.1	12.0	32
2.21	5.8	82.9	10.4	56

A noteworthy characteristic of the slag was its decrepitating property. During the smelting operation lower titanium oxides may have formed, which decomposed on cooling, causing the decrepitation. Observations indicated that the higher the  $\text{TiO}_2$  content, the higher the temperature at which this phenomenon began. Figure 5 is a view of a pile of slag cones in various stages of decrepitation.

#### Recovery and Grade of Products

Virtually all of the titanium, over 90 percent of the manganese, and most of the columbium were recovered in the slag product. The slag consistently contained 80 percent or more  $\text{TiO}_2$ ; occasionally it contained as much as 86 percent. Unfortunately, when this grade was produced the slag was so viscous that it became necessary to reduce the amount of reductant in the furnace charge to maintain continuous operations.



Figure 5. - High-titanium slag decrepitating on cooling.

The  $TiO_2$  content of slag produced from Idaho ilmenite is higher than that of all commercially available titanium raw materials except rutile. Table 6 shows comparative analyses of rutile, Sorel slag, Florida ilmenite, and slag produced from Idaho ilmenite.

TABLE 6. - Typical analyses of various titanium raw materials, percent

	Commercial rutile	Albany slag		Sorel slag	Florida ilmenite
		Low Mn	High Mn		
$TiO_2$ .....	97.0 - 98.5	83.9	83.4	71.7	64.1
$FeO$ .....	.1 - .5	10.7	3.9	9.08	4.9
Fe (total) .....	-	7.3	3.0	7.28	21.8
$MnO$ .....	-	5.75	9.1	.41	1.66
$MgO$ .....	-	1.07	1.03	5.29	.35
$CaO$ .....	-	.1	.1	.94	.13
$SiO_2$ .....	.1 - 1.0	1.48	1.68	5.10	.20
$Al_2O_3$ .....	.05- .1	.56	.64	6.70	1.24
$ZrO$ .....	0 - 1	-	-	-	-
$Cb_2O_5$ .....	.3 - .6	.53	.22	-	-
$V_2O_5$ .....	.3 - 1.0	-	-	-	.13
Cr .....	.2 - .4	-	-	-	-
Sn .....	0 - .2	-	-	-	-
$P_2O_5$ .....	-	-	-	.02	.22
$Cr_2O_3$ .....	-	-	-	.41	.09

Table 7 shows spectrographic analyses of 3 lots of slag and 1 of pig iron. The analyses indicate that the  $MgO$  content of the slag was slightly higher when the ilmenite was smelted in the magnesite-lined furnace than in the carbon-lined furnace.

TABLE 7. - Spectrographic analyses of typical slag and pig iron products

Sample	Al	As	Ca	Cu	Mg	Fe	Mn	Si	Ti	V
Slag, from lot 1 ilmenite .....	D	D	E	F	G	A	C	C	A	E
Slag from lot 6 ilmenite .....	D	D	D-	F	D+	A	C	D+	A	D
Slag from lot 5 ilmenite .....	D	-	-	F	D	A	A	C	A	E
Pig iron from lot 1 ilmenite .....	ND	D	E	F	G	A	E	F	F	F

Legend: A - over 10 percent  
B - 5 to 10 percent  
C - 1 to 5 percent  
D - 0.1 to 1 percent

E - 0.01 to 0.1 percent  
F - 0.001 to 0.01 percent  
G - under 0.001 percent  
ND - none detected

From 75 to 85 percent of the iron charged to the smelting furnace was recovered in the metal product. The pig iron was highest in carbon when smelted in the carbon-lined furnace. The manganese content was highest during the period manganese ore was used in the furnace charge. The pig iron is of acceptable quality for most uses. Analyses of some of the pig iron products are shown in table 8.

TABLE 8. - Typical analyses of pig iron products, percent

Sample	Fe	C	Mn	Ti	P	S
Metal produced in carbon-lined furnace .....	94.2	4.57	0.09	0.09	0.29	0.45
Metal produced in magnesite-lined furnace ....	-	3.46	.09	.06	.22	.08
Metal produced when using Mn ore in charge ...	-	2.28	.57	.08	.24	.07

### Electric Power and Electrode Requirements

The electric power required to smelt ilmenite is considerably higher than that required to recover an equivalent amount of pig iron from iron ore because the smelting operation is conducted at a higher temperature and the slag-to-metal ratio is higher. The electric power consumption varied from 4,417-5,884 kw.-hr. per ton of pig iron recovered. The electric power consumption was generally highest when producing titanium slag highest in  $\text{TiO}_2$ , as the highest smelting temperatures were attained under these conditions. The slag-to-metal ratio averaged approximately 2. When smelting a good grade of iron ore in the same furnace, the electric power consumption was approximately 2,400 kw.-hr. per ton of pig iron recovered, the slag-to-metal ratio was about 0.5, and the tapping temperatures were approximately 300°C. lower.

The electrode consumption is a function of the current used in an electric smelting operation. A lower voltage is required when smelting ilmenite than is required in most smelting operations because of the higher electrical conductivity of the titanium slag. Therefore, the electrode consumption can be expected to be higher. The electrode consumption for all but one test was indicated to be between 14.1 and 22.7 pounds per ton of ilmenite smelted. The electrode consumption of 6.3 pounds per ton of ilmenite smelted, reported for the other test appears to be too low to be considered.

### CONCLUSIONS

These smelting tests indicate that it is feasible to produce a slag containing over 80 percent  $\text{TiO}_2$  and a marketable pig iron by the electric smelting of Idaho alluvial ilmenite concentrates. Probably the slag has its highest potential value as a raw material in producing titanium tetrachloride for use in the titanium metal industry.

Smelting research should be continued to determine whether smelting temperatures can be increased so as to produce a slag of higher titanium content, and to investigate further the use of manganese in the charge for displacing iron from the slag.

The electric power consumption is considered reasonable. If all power used in the ilmenite-smelting operation is charged to the pig iron, the energy requirement is little more than twice that required to produce pig iron from a good grade of iron ore.

The graphite electrode and magnesite refractory consumption is not considered excessive for this type of an operation.