

[54] **PROCESS FOR REDUCING IRON CONTENT OF TITANIFEROUS MATERIAL**

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[22] Filed: **Aug. 29, 1972**

[21] Appl. No.: **279,110**

**Related U.S. Application Data**

[63] Continuation of Ser. No. 60,124, July 31, 1970.

**Foreign Application Priority Data**

July 31, 1969 United Kingdom..... 38475/69

[52] U.S. Cl..... **75/1; 75/26**

[51] Int. Cl..... **C22b 1/10; C22b 53/00**

[58] Field of Search..... 60/124, 127, 123; 75/1 T, 75/26

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[57]

**ABSTRACT**

A process for the beneficiation of ironcontaining titaniferous material in which, during one stage of the beneficiation, the iron content of the material is reduced with hydrogen in the presence of added steam to control the rate of reduction.

**11 Claims, No Drawings**

## PROCESS FOR REDUCING IRON CONTENT OF TITANIFEROUS MATERIAL

This is a continuation of application Ser. No. 60124, filed July 31, 1970.

The present invention relates to the reduction of iron-containing materials, particularly to the reduction of iron-containing titaniferous materials.

In some processes for the beneficiation of iron-containing titaniferous material such as ilmenite or leucoxene it is advisable to reduce the iron content substantially wholly to the ferrous state while avoiding the formation of significant amounts of metallic iron or trivalent titanium. For example, a suitable product from such a process is one which contains not more than 2 percent of the iron content in a form other than the ferrous state.

In the past the control of the reduction at elevated temperature to produce such a product has been found difficult.

For example, when hydrogen is used the reduction is generally so rapid that it is difficult to avoid the formation of unacceptable amounts of metallic iron and/or trivalent titanium. When a solid reductant such as carbon is used, on the other hand, it has been found that at temperatures up to about 1,000°C the reduction is unacceptably slow whereas at higher temperatures it is very rapid and gives rise to excessive amounts of metallic iron.

One method by which the foregoing difficulties may be avoided is by the use of hydrocarbon containing from 1 to 4 carbon atoms, particularly methane, as a reductant.

It is an object of the present invention to provide another method whereby these difficulties may be overcome.

Accordingly, the present invention is a process for reducing the iron content of iron-containing titaniferous material by contacting the material at an elevated temperature with hydrogen in the presence of added steam.

The iron-containing titaniferous material is normally an ilmenite or leucoxene and preferably the former. Such materials may have an iron content in the range of about 5 to 55 percent and more commonly one in the range 30 to 46 percent by weight (expressed as FeO).

The iron-containing titaniferous material is preferably contacted with the reducing agent when in finely-divided form, for example when having an average particle size in the range 50 $\mu$  to 300 $\mu$  and preferably one in the range 100 $\mu$  to 200 $\mu$ .

The material may be contacted with the reductant in any suitable equipment but it is preferred to carry the reaction out in a fluidised bed or in a rotary kiln.

Where a fluidised bed is used the flow rate of the gas through the bed may vary from the minimum fluidising velocity to one at which excessive carry over from the bed commences. Preferably the flow rate is in the range 5 to 15 times the minimum fluidising velocity of the bed.

It may be advisable to incorporate an inert gas such

as nitrogen, argon or carbon dioxide into the hydrogen/steam mixture, for example to ensure fluidisation and/or to assist in controlling the rate of reaction.

Temperatures at which the iron-containing titaniferous material may be contacted with hydrogen and steam (and other gas if present) are suitably in the range 600°C to 1,100°C but are more preferably in the range 650°C to 950°C and particularly in the range 700°C to 900°C.

The process is conveniently carried out in a continuous manner, i.e. in which reduced material is continuously or intermittently removed from, and unreduced material is continuously and intermittently added to, the reaction zone.

The proportion of steam added to the hydrogen will determine the degree of reduction which takes place. For example, when using hydrogen without the introduction of steam both metallic iron and trivalent titanium are normally formed as well as ferrous iron if the reduction period is prolonged. As the proportion of steam introduced is progressively increased, say up to 10 percent by volume on the hydrogen present, the amount of metallic iron formed falls (and generally becomes constant at a very low figure) while the amount of ferrous iron (as opposed to ferric iron) may also fall slightly. It is preferred, therefore, to add an amount of steam which gives a product with a very small proportion of metallic iron and a very high proportion of ferrous iron. Proportions of about 1 to 7 percent steam are therefore preferred, particularly proportions in the range 2 percent to 5 percent, by volume on the hydrogen present.

The process of the present invention is particularly suitable for reducing iron-containing titaniferous material which has been subjected to a preliminary oxidation step and/or which will subsequently be subjected to a leaching step, for example with a mineral acid such as aqueous hydrochloric acid to remove the reduced iron. In the latter case the comparative absence of trivalent titanium and ferric salts is of particular value if the solution is to be subsequently treated to recover finely-divided iron or iron oxide and/or to recover hydrogen chloride, for example, by the Aman process.

The following Example shows various embodiments of the process of the present invention.

### EXAMPLE

A beach sand ilmenite containing 39.5 percent by weight of iron (expressed as FeO) was oxidised at 875°C until substantially all the iron was in the ferric state.

Equal quantities of the material were then placed in turn into a 3 inch diameter bed and the bed was heated. When the temperature reached 700°C the bed was fluidised with hydrogen and with mixtures of hydrogen and steam at a flow rate five times the minimum fluidising velocity for the times shown in Table 1 while maintaining the temperature of 700°C.

The materials were analysed after reduction for 60 minutes and the results are given in Table 2.

TABLE I

Nominal % v/v H <sub>2</sub> O	H <sub>2</sub> flow rate l/min. at ambient temperature	H <sub>2</sub> O flow ml. of liquid per minute	% FeO* after minutes											
			5	10	15	20	25	30	35	40	45	50	55	60
0	10.85	—	18.0	29.6	32.7	36.2	38.6	39.4	39.1	40.6	—	42.2	—	41.1
2	10.19	0.156	16.1	29.8	33.3	34.9	36.4	37.0	37.9	39.0	39.9	39.0	39.9	39.8
5	9.2	0.364	16.4	29.6	31.5	34.6	35.6	36.0	36.6	37.1	37.8	37.6	36.9	38.0
10	8.1	0.675	11.9	28.2	31.0	33.9	34.5	34.8	34.1	35.5	35.3	35.9	35.6	36.8

Table 2

	Analysis of bulk sample after 60 minutes		
	% metallic Fe	% Ti <sub>2</sub> O <sub>3</sub>	% FeO*
0	0.5	7.4	41.1
2	0.3	1.1	39.8
5	0.2	0.4	38.0
10	0.2	0	36.8

In the FeO determination, metallic iron and part of the trivalent titanium will be determined as FeO. This explains the higher levels when hydrogen only is used as reducing agent.

Note:

The apparent excess of iron (expressed as FeO) after reduction with hydrogen over that of the starting material is due to the presence of metallic iron and some trivalent titanium (which are estimated as FeO by the method of analysis).

What is claimed is:

1. A process for reducing the iron content containing titaniferous material comprising oxidising said titaniferous material until substantially all the iron content is in the ferric state, and contacting said oxidised material in a fluidised bed at a temperature within the range 650°C. to 950°C. with a mixture of hydrogen and added steam until not more than 2 percent of the iron content of said material is in a form other than the ferrous state, the proportion of steam in the mixture being in the range of about 2 to 5 percent by volume of hydrogen.

2. A process as claimed in claim 1 wherein the iron-containing titaniferous material is ilmenite or leucoxene.

3. A process as claimed in claim 1 wherein the iron-containing titaniferous material has an iron content in the range 30 to 46 percent by weight as expressed as FeO.

4. A process as claimed in claim 1 wherein the iron-containing titaniferous material has an average particle size in the range 50μ to 300μ.

15 5. A process as claimed in claim 4 wherein the average particle size is in the range 100μ to 200μ.

6. A process as claimed in claim 1 wherein an inert gas is added with the hydrogen and steam.

20 7. A process as claimed in claim 1 wherein the material is heated to a temperature in the range 700°C to 900°C.

8. A process as claimed in claim 1 wherein the reduced material is leached with a mineral acid to remove iron from the titaniferous material and solid beneficiated titaniferous material is separated from the suspension.

25 9. The process of claim 1 wherein in addition to said added steam there is also incorporated an inert gas selected from the group consisting of nitrogen, argon and carbon dioxide to ensure fluidisation.

30 10. The process of claim 9 wherein said titaniferous material is contacted with said mixture in a fluidised bed, the flow rate of gases through said bed being in the range five to 15 times the minimum fluidising velocity of said bed.

35 11. The process of claim 1 wherein said titaniferous material is contacted with said mixture in a fluidised bed, the flow rate of the gases through said bed being in the range five to 15 times the minimum fluidising velocity of the bed.

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 3,877,929 Dated April 15, 1975

Inventor(s) Whitehead et al

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Under the heading "Inventors", after "Stockton-on-Tees," the phrase "both of England" should be deleted and the phrase -- ; Alan Conners, Delamere, Stockcroft Road, Balcombe, Sussex; and Derek Vernon Gosden, Reksley House, Shipley Road, Southwater, Horsham, Sussex, all of England -- should be inserted.

Between the headings "Inventors" and "Filed", the heading and phrase -- Assignee: British Titan Limited, Billingham, Teesside, England -- should be inserted.

Signed and Sealed this

thirteenth Day of April 1976

[SEAL]

Attest:

RUTH C. MASON  
Attesting Officer

C. MARSHALL DANN  
Commissioner of Patents and Trademarks