

UNITED STATES PATENT OFFICE.

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ORS TO THE TITANIUM ALLOY MANUFACTURING COMPANY, OF NEW YORK, N. Y.,
A CORPORATION OF MAINE.

METHOD FOR CONCENTRATING TITANIC OXID.

1,106,407.

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No Drawing.

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To all whom it may concern:

Be it known that we, AUGUSTE J. ROSSI and LOUIS E. BARTON, both citizens of the United States, and residents of Niagara Falls, in the county of Niagara and State of New York, have invented certain new and useful Improvements in Methods for Concentrating Titanic Oxid, of which the following is a specification.

Uses of titanic oxid in the arts are now extensive and increasing. A long standing problem has been and still is its accumulation, on industrial scales of manufacture, in sufficient quantity and purity, and at the least possible cost.

Numerous known substances contain greater or less percentages of titanic oxid (TiO_2) in combination with other metallic oxids. Natural titanic oxid is, as well known, widely and plentifully existent in ores, but, almost invariably, so entangled, or combined, with other oxids and constituents of the gangue, as to render it, as mined, unavailable for most industrial purposes. As thus found, in varying percentages, in enormous existing deposits of so-called "titaniferous-iron," or "ilmenorutile," ores, it is particularly associated with corresponding percentages of iron oxids, from which it has hitherto proved practically impossible, without prohibitory expense, to so completely separate it as to render it available for many desired industrial uses. Even the purest, so-called "rutile," ores, though of much higher titanic oxid content than any others, as high for example as 95%, contain nevertheless percentages of iron oxids, silica and earthy bases, such as alumina, rendering them, in natural state, unsatisfactory contributors of titanic oxid in the manufacture of certain special products, and, moreover, deposits of such "rutile" ores have hitherto been discovered only in segregated quantities too small to supply existing industrial demands. Moreover the said ores usually contain more or less "ilmenite" being a compound of titanic oxid and iron oxid designated by the formulas FeTiO_3 , or FeO TiO_2 , which compounds we believe to have hitherto proved too refractory for any commercially practicable method of deriving titanic oxid from such last mentioned ores.

The objects of our present invention comprise production of methods whereby titanic oxid may be more completely separated from other constituents, including particularly the iron oxids, of said substances or ores, and this more rapidly, simply, and economically, than heretofore. We attain these objects by our present improved method as follows:—We first finely pulverize the ore and then smelt it together with oxid of an alkali metal (preferably a hydroxid, such as commercial caustic soda) and a reducing agent, preferably carbon in some form, until the charge is thoroughly melted and iron oxids present reduced to molten elemental iron. The relative proportions of the said constituents of the charge are preferably such as, determined by analysis of the particular substance treated, to supply sufficient of said oxid of an alkali metal to combine with elements and compounds present having chemical affinity therefor, as for example with the titanic oxid to form titanate of soda (Na_2TiO_3), and sufficient carbon to reduce the iron oxid, or oxids, present. But neither some deficiency, nor some excess, is prohibitory. For example merely,—in treating an ore which analyzed—

Titanic oxid	52	
Iron oxids	40	
Silica	3	
Alumina	4	85

we successfully proportioned the charge as follows, viz:—

Ore	298 parts	—59.6%	
Caustic soda (70% Na_2O)	172 "	—34.3%	90
Coke	30 "	—6.0%	

The smelting of the charge is preferably conducted in an electric furnace, but it may of course be accomplished by any other desired means capable of insuring a complete melt and the reduction of the iron oxids to molten iron, as for example in an open hearth furnace heated by a direct coke fire in a grate, or indirectly by producer gas, such furnaces being of course lined with material sufficiently refractory to resist the resulting reactions.

We believe that, during the melt, the fol-

lowing reactions, among others, occur in the charge, viz:—Oxids of iron present are reduced to elemental iron. Oxids of iron, if any, remaining unreduced to molten iron, are maintained in ferrous state by action of the carbon. Oxid of sodium combines with oxid of titanium to form titanate of soda, thus readily breaking up ilmenite molecules present. Silica and alumina of the gangue combine with the oxid of sodium to form silicates and aluminates of soda.

After the reactions are completed, the temperature is maintained sufficiently to enable the resulting iron to segregate, as by percolating through the charge, and to concentrate in an underlying molten bath, the which is then separated from the supernatant remainder, or melt, as by tapping and casting it, after which the remaining melt is tapped out separately. The resulting pig iron constitutes a valuable by-product of our process possessing, owing to its origin, exceptional purity and toughness. Of course other methods of separating the elemental iron from the melt might be resorted to without departing from our invention, though at undesirably greater cost and with resulting products inferior for our purposes. The remainder of the melt, after withdrawal therefrom of the elemental iron, is, in well crushed condition, say to about 80 mesh, repeatedly and thoroughly lixiviated in heated, preferably boiling, water, until substantially all the said therein contained titanate of soda has been decomposed into titanic oxid and caustic soda. The soda thus set free, together with any excess soda and said silicates and aluminates, being soluble, dissolve, and are thus removed from the then remaining melt with the water of lixiviation. It will be noted that the soda lye resulting from said lixiviation can be evaporated to a cake of soda useful in treating as aforesaid the next charge of ore. In our practice we have thus repeatedly recuperated approximately 85%, or even more, of the soda employed, the which constitutes an important economy in the practice of our present method.

What still remains of the melt after its aforesaid treatment may be expected to be substantially free from excess of soda, from silica and alumina and their compounds, and from iron oxids, but to retain substantially all of the titanic oxid and possibly some small quantities of unreduced iron oxids, these being insoluble in water, also some slight remainders of gangue, including magnesia, the ores treated containing not to exceed traces of lime. It will be noted that the said unreduced iron oxids, being as aforesaid in ferrous state, are more readily soluble in the hereinafter mentioned acid bath, than if in ferric state. Those portions of the melt which are insoluble in

water we then subject, and preferably while still moist, to the action of a heated, preferably boiling, bath containing an iron solvent acid, so diluted as to dissolve therein ferro, and other, compounds, excepting the titanic oxid. We prefer to use, for this purpose sulfuric acid, owing to the latter's comparative low cost and superior effects, though other suitable acids, such as hydrochloric for example, may be employed with useful results, if diluted so as to operate as above specified. In this step of our method we have, with excellent results, proportioned the water of the bath to be from three to four times the weight of the therein heated melt, and the acid, for example sulfuric acid, in proportion of from about 29 parts to each 20 parts of said melt. Though, by the proportions last mentioned, the sulfuric acid is in considerable excess, it is not wasted, since it may be withdrawn, or decanted, to be used, with or without addition of fresh acid, in treatment of the next charge. This last mentioned treatment of the melt we continue for about an hour, by which time the titanic oxid, the which remains undissolved in the bath, will have settled to the bottom, and, being separated from the latter, as by decantation, these settlings will, if our method has been properly followed as above, be found, after calcining them, to consist of pure titanic acid in proportion as high as from say 96 to 97%.

We are thus enabled to produce, from ordinary titaniferous-iron or ilmeno-rutile ores, and, as we believe, by a more rapid, simple, and economical process than heretofore, a titanic oxid product higher in its percentage of that compound than are the best and rarest of known rutile ores, and this at a cost very greatly below the present market price of such last mentioned ores.

In the practice of our aforesaid method, potassium hydroxid (KOH) can be used as a substitute for the caustic soda particularly described. In fact, as above stated, the hydroxid of any other alkali metal may, if desired, thus be used. We prefer however caustic soda because of its comparative cheapness and, as we believe, superior efficiency under the conditions involved.

It will be noted that the distinguishing characteristics of our present method comprise substantial separation from the ore of its ferro constituents by withdrawing them in molten state as reduced to elemental iron, and also the maintenance of substantially all the titanic oxid throughout the operations in undissolved state.

While we have particularly referred, throughout our preceding specification, to ores, we do not wish thereby to confine the application of our methods to ores specifically, it being understood that such application to any other substance, or material,

containing titanic oxid and iron oxid is also feasible and within the purview of our invention and claims.

What we claim as new and desire to secure by Letters Patent is the following, viz:—

1. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with oxid of an alkali metal in presence of a reducing agent; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to the action of an iron-solvent acid so diluted as to dissolve therein ferro, and other compounds excepting the titanic oxid.

2. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with oxid of an alkali metal in presence of a reducing agent; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to the action of a bath containing dilute sulfuric acid.

3. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with oxid of an alkali metal in presence of carbon; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to the action of a bath containing an iron-solvent acid so diluted as to dissolve therein ferro, and other compounds, excepting the titanic oxid.

4. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with oxid of an alkali metal in presence of carbon, withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to the action of a bath containing dilute sulfuric acid.

5. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with sodium hydroxid in presence of a reducing agent; withdrawing iron from the resulting melt; lixiv-

ating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to a bath containing an iron-solvent acid so diluted as to dissolve therein ferro, and other, compounds, excepting the titanic oxid.

6. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with sodium hydroxid in presence of carbon; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to a bath containing an iron-solvent acid so diluted as to dissolve therein ferro, and other, compounds excepting the titanic oxid.

7. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with sodium hydroxid in presence of a reducing agent; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to a bath containing dilute sulfuric acid.

8. The method of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, which comprises smelting said substance together with sodium hydroxid in presence of carbon; withdrawing iron from the resulting melt; lixiviating the remainder of said melt in water; withdrawing the insoluble portion and subjecting it to a bath containing dilute sulfuric acid.

9. In the process of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, the step which consists in smelting said substance together with oxid of an alkali metal and carbon therewith intermixed substantially as and for the purpose described.

10. In the process of concentrating titanic oxid (TiO_2) from a substance containing it and iron oxid, the step which consists in smelting said substance together with sodium hydroxid and carbon therewith intermixed substantially as and for the purpose described.

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Witnesses:

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