UNITED STATES PATENT OFFICE.

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PROCESS FOR THE RECOVERY OF TITANIUM FROM ITS ORES.

1,357,690. Specification of Letters Patent. Patented Nov. 2, 1920.

No Drawing. Application filed December 6, 1918. Serial No. 265,494.

To all whom it may concern:

Be it known that I, OSCAR T. COFFELT, a citizen of the United States, residing at Chicago, county of Cook, and State of Illinois, have invented certain new and useful Improvements in Processes for the Recovery of Titanium from Its Ores, of which the following is a specification.

My invention relates more particularly to the separation of titanium from the ores in which it is found associated with iron in the form of the oxid. One of the most common of such ores is ilmenite which contains about fifty per cent. (50%) of titanium oxid and the process will be described more particularly with reference thereto. My improved process comprises two principal operations or steps in the first of which the ore is digested with sulfuric acid by means of which the titanium and iron contained in the ore are brought into solution as sulfates. From this solution the titanium is crystallized out as a double salt of titanium in which form it may be marketed, or it may be further converted into the hydroxid by known methods.

In the preferred practice of my invention the ilmenite is ground to a degree of fineness in which it will pass through a 200 mesh screen. It is then digested with sulfuric acid by which the titanium and iron present are converted into the sulfates. Previous to my invention concentrated sulfuric acid of commercial strength (about 95%) has been employed for this purpose. With acid of this strength, however, the reaction is comparatively slow and certain other changes take place whereby the yield of titanium sulfate is diminished. I have discovered, however, that a concentration of sulfuric acid of about 81½ per cent. is most effective, the action being more complete and rapid with this concentration than with concentrations either substantially higher or lower. Very good results are obtained, however, with concentrations running anywhere from 75 to 90 per cent. Acid of lower concentration than that indicated can be used and will become more concentrated during the progress of the digestion by distillation. But the reaction in such case only becomes lively as the concentration approaches the percentages indicated. Preferably about three times as much sulfuric acid (figured as concentrated) by weight is used as the ore to be treated, though this precise proportion is not essential. The digestion takes place preferably at a temperature of about 160° C., though a somewhat higher or lower temperature may be employed. Ordinarily the digestion starts at a lower temperature and finishes somewhat higher. However, it is preferably maintained at about that indicated until the process is completed. Substantially all the titanium and iron in the ore is now in the form of the sulfate and may be separated from the insoluble matter by introducing a sufficient quantity of water to bring the gravity of the solution to 35° to 40° Baumé. Thus where fifty pounds of ilmenite have been digested with one hundred and fifty pounds of acid (figured as above) the digested mass may be introduced into twenty-five gallons of water. The solution is heated for the purpose of insuring that all of the soluble material is taken up. After cooling the solution is decanted and filtered or otherwise freed from the insoluble residue.

The titanium is next separated from the iron. A number of methods are known for accomplishing this. A preferred method not known prior to my invention thereof is by adding a reagent which will throw the former out of solution but leave the latter dissolved. For this purpose preferably sodium chlorid, (common salt) is employed as being efficient and inexpensive. For the most complete separation the solution is concentrated by evaporation either before or after the addition of the salt. If before, the solution is brought to a gravity of about 53° B. Preferably the salt is added in solid form to the solution after it has been concentrated. Where sodium chlorid is the reagent employed in the separation, I preferably use an amount approximating .6 or .7 times the mass of the sulfuric acid (calculated as 100 per cent. acid) which has been employed for the digestion. After the sodium chlorid is added the solution is further heated until the salt is dissolved. During this operation the titanium begins to crystallize out as sodium titanium sulfate, the amount increasing as the volume of the liquid diminishes by evaporation.

After the above concentration is reached the solution is permitted to cool and as the temperature falls crystallization of the sodium titanium sulfate continues until, when

ordinary temperatures are reached, the titanium salt has practically completely crystallized. The iron in the solution has in the meantime been largely converted into the chlorid and, due to the presence of the chlorid iron, the chlorid and sulfate are more soluble than the titanium sodium sulfate and remain practically in solution until the latter is entirely separated. If it is desired to separate the sodium titanium sulfate quantitively the crystallization may be carried to a point at which a small proportion of the iron salt has also crystallized the complete separation of the latter being reserved for a subsequent operation.

After the crystallization of the titanium salt has been carried sufficiently far a small amount of water is added to the mass so as to cause solution of any iron which has crystallized out. The crystalline sodium titanium sulfate is then separated from the liquor containing the iron salt by filtration or otherwise.

Where it is desired to further purify the product this may be effected by solution and recrystallization as follows: A 30% sulfuric acid solution is used to dissolve the sodium titanium sulfate, the solution being heated for this purpose, and then evaporated down to about 45° B. as before. Sodium chlorid is then added and the solution further heated until the double salt has crystallized out.

The sodium titanium sulfate is of itself a commercial product and may be marketed in that state. If desired, however, it may be first converted into the hydroxid in a well known method by the use of soda ash or caustic soda.

As above indicated, the process is not confined to the treatment of ilmenite but is of great value in connection with titanium oxids in which iron is found to be present. Common salt has been more specifically referred to as the separating reagent more particularly because of its cheapness. But chlorids of the other alkalis (including ammonium) may be substituted in whole or part therefor. A mixture of chlorids or chlorids and sulfates may be used instead of the chlorid if preferred. If the potassium salt is used the double sulfate only crystallizes out on cooling and when the ammonium salt is used the crystallization takes place at a lower temperature than when the sodium chlorid is employed.

I claim:

1. A method of separating titanium from ores thereof containing iron, which consists in digesting the ores with sulfuric acid of a strength between 77% and 85%, thereby forming a solution of iron and titanium sulfates, and depositing the titanium from said solution.

2. A process of separating titanium from its ores containing iron, which consists in digesting the ore with sulfuric acid of approximately 81½% strength, and then depositing the titanium from the solution so formed.

3. A method of separating titanium from an acid solution thereof which contains iron, which consists in adding to the solution an alkali metal salt of a halogen acid, and then crystallizing out the double salt of titanium and an alkali formed.

4. A process of separating titanium from a solution containing iron and titanium, which consists in adding to the solution the chlorid of an alkali metal and then crystallizing the double titanium salt so formed.

5. A process of separating titanium from a solution containing iron in admixture which consists in adding to said solution chlorid of sodium and crystallizing the double salt of sodium and titanium so formed.

6. A process of separating titanium from a solution containing titanium and iron as sulfates, which consists in adding sodium chlorid thereto and crystallizing the sodium titanium sulfate so formed therefrom.

7. The process of separating titanium from a solution containing the same and iron as sulfates, which consists in concentrating the solution, adding an alkali metal chlorid thereto, and evaporating the solution to crystallize out the double salt of titanium and the alkali.

8. A process of separating titanium from a sulfuric acid solution containing iron, which consists in concentrating the solution, adding sodium chlorid thereto, and crystallizing by evaporation until the titanium sodium sulfate is substantially separated.

9. A process of separating titanium from its ores containing iron, which consists in digesting the ores with strong sulfuric acid at a temperature of approximately 160° C., separating the solution of titanium and iron sulfates so formed from the insoluble matter, adding chlorid of sodium to the solution, concentrating the solution to crystallize out the double salt of sodium and titanium, and separating the crystalline deposit from the liquor.

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