UNITED STATES PATENT OFFICE.

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PROCESS OF PRODUCING TITANIUM NITROGEN COMPOUNDS.

1,408,661. Specification of Letters Patent. Patented Mar. 7, 1922.

No Drawing. Application filed October 25, 1920. Serial No. 419,459.

To all whom it may concern:

Be it known that we, FOORD VON BICHOWSKY and JOHN HARTHAN, citizens of the United States, and residents of Glendale, county of Los Angeles, State of California, have invented a new and useful Improvement in Processes of Producing Titanium Nitrogen Compounds, of which the following is a specification.

The present invention relates to a process of producing titanium nitrogen and the like compounds from such minerals as ilmenite and one of the important features of the invention resides in the recovery or production of such compounds by heating ilmenite with an agent such as carbon and in the presence of an agent such as nitrogen. We have discovered that titanium oxide contained in such minerals as ilmenite ($FeO.TiO_2$) or other titaniferous iron ores rich in titanium, can be readily converted into the nitrides, or the so called cyanonitride of titanium by heating fine particles of the mineral; with an oxygen containing salt of an alkali metal and carbon while passing nitrogen or impure mixtures of nitrogen, carbon dioxide, monoxide, methane and the like free from such other gases as sulphureted hydrogen which would harmfully combine with the constituents of the heated mass; at the temperatures slightly below that at which the treated mass would melt.

The oxygen salt of the alkali metal may be either an oxide or hydroxide of sodium or potassium, or a carbonate or formate or even a silicate or titanate of those metals or mixtures of the above or any of their equivalents. Salts which like the sulphates would be reduced and form harmful compounds such as iron sulphide should not be employed.

The carbon may be employed for instance, in the form of soot, or coke, or of a compound containing carbon, such for instance as crude oil, and instead of these, or in addition thereto, gases capable of yielding carbon, such as acetylene or methane or mixtures of the latter occurring as natural gas, may be passed over the heated mixture. By this procedure the iron is reduced and saturated with carbon while the alkali metal salt serves, together with the iron to catalytically accelerate the combination of the titanium with the nitrogen. From the mixture of iron and titanium nitride the iron is easily removed and the titanium nitride recovered.

A process based on the present invention is particularly valuable since the by-products, which may be obtained, have a wide commercial value and as a result the titanium nitride can be obtained at a remarkably low figure. Thus enabling use to be made of it directly as an artificial fertilizer or it may be employed as a source of pure titanium dioxide or other titanium salts.

Having now defined the purpose of our discovery the following examples will serve to illustrate further the nature of our invention and the preferred method of carrying it into practical effect, however it is not confined to these examples as various departures may be found to bring about the desired result. The parts by weight are about as follows:

100 parts ilmenite ground to pass 100 mesh, to the inch, screen, 5 parts anhydrous soda ash, 10 parts pitch, the whole worked and made into briquettes by any suitable method. These briquettes are then heated for a period of about six hours during which time they are subjected to methane or natural gas under a slight pressure and containing about 20% by volume of nitrogen. At first, the temperature may be between 1100–1200° C. but finally the temperature should be maintained between 1000–1100° C. to prevent the reduced iron, which by this time is saturated with carbon, from melting. The titanium nitride iron mixture so obtained is allowed to cool in an atmosphere of a neutral gas and when cold the iron is dissolved out with warm dilute sulphuric acid. The ferrous sulphate solution, so obtained, is filtered and crystallized. The titanium nitrides or cyanonitride or mixtures of both can be used for the preparation of ammonia or directly as a fertilizer.

As another example:

100 parts ground ilmenite, 10 parts crude oil tar briquetted and then heated in a stream of natural gas at about 1000° C., for three hours or until the iron is greatly reduced. Then allowed to cool and ground up and by the addition of 10 parts iron filings, 5 parts soda ash, 5 parts water glass 42° Bé., 10 parts water, the whole is well mixed and pressed into briquettes. Upon heating these

briquettes in a methane nitrogen mixture for about three or more hours at 1000–1050° C., the iron is converted into impure iron carbide and the titanium oxide into nitrides. Upon treating this resulting product as in our application filed October 25, 1920, Serial Number 419,458 sodium cyanide is formed.

The improved process of thus producing sodium cyanide is not claimed as a part of the present invention but is divided and covered in a co-pending application.

We claim:

1. The process of producing titanium nitrogen compounds which consists in heating ilmenite and oxygen salt, other than a thio acid of an alkali metal, in lesser quantity than would be required to convert all of the ilmenite into a titanate, with carbon in the presence of nitrogen.

2. The process of producing titanium nitrogen compounds which consists in heating ilmenite with carbon and sodium carbonate in the presence of nitrogen.

3. The process of producing titanium nitrogen compounds which consists in heating ilmenite with carbon and sodium carbonate in the presence of nitrogen at a temperature to avoid fusing of the mass.

4. The process of producing titanium nitrogen compounds which consists in heating ilmenite with carbon, sodium carbonate and nitrogen at a temperature of about 1100° C.

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