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PREPARATION OF METALLIC TITANIUM

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This invention relates to the preparation of titanium metal, more particularly to production of the metal by reduction of titanium chloride by reaction with an alkali metal.

In methods heretofore proposed for reacting titanium chloride with alkali metals, difficulty has been experienced in that the alkali metal becomes coated with a layer of alkali metal chloride, which prevents complete utilization of the alkali metal. The resulting reaction product is a mixture of titanium metal, alkali metal and salt, and the presence of the unreacted alkali metal adds to the difficulty in working up this material to obtain pure titanium.

An object of the present invention is to provide an improved process for reducing titanium chloride or other titanium halide by reaction with an alkali metal. Another object is to reduce a titanium halide with an alkali metal in such manner as to obtain directly a product substantially free from alkali metal and to obtain substantially complete utilization of the alkali metal in the reduction reaction. Further objects will be apparent from the following description of the invention.

The above objects may be attained in accordance with the present invention by providing a layer of liquid alkali metal floating on a bath of fused salt and passing a titanium halide in liquid or preferably in vaporous state into the bath below the layer of alkali metal. The temperature of the bath and alkali metal is maintained at above the boiling point of the titanium halide and the introduction of the titanium halide preferably is continued until the alkali metal is substantially completely consumed. The surface of the alkali metal may be protected from oxidation by enclosing it in an inert atmosphere, e. g., hydrogen or nitrogen. By this method I am able to readily obtain substantially complete reaction of the alkali metal, obtaining at the end of the reaction, a mixture of titanium and salt. The titanium may be recovered by washing out the salt by conventional methods.

As an illustration of my invention, titanium metal may be prepared by reacting titanium tetrachloride vapor with molten sodium supported on a molten salt bath, the melting point of which lies below the boiling point of sodium. The salt bath may consist of an alkali metal chloride or mixture of alkali metal and alkaline earth metal chlorides. I prefer to use a bath of fused potassium chloride and operate with a bath temperature of 700 to 800° C. The sodium chloride formed in the reaction mixes with the bath to form a low-melting eutectic mixture. The titanium tetrachloride vapor is passed into the salt bath, below the layer of sodium and preferably the mixture is agitated by a suitable stirring device during the reaction. The process is carried out in an enclosed space, with an atmosphere of hydrogen or other suitable inert gas over the sodium layer.

The invention is further illustrated by the following example.

Example

Six kg. of potassium chloride and 10 kg. of sodium are placed in a cylindrical covered iron vessel, from which the air is displaced by hydrogen and heated to fusion. When the temperature of the melt has reached about 700° C., 20.6 kg. of titanium tetrachloride is introduced by way of an inlet tube leading into the lower portion of the melt. During the reaction, the layer of sodium floating on the molten salt is agitated by a stirring device. The temperature of the reaction mixture is so regulated that up to the end of the reaction the temperature in the salt bath does not exceed 900° C. When the reaction is completed, the mass is cooled and titanium metal is recovered by leaching with water to remove the salt. By further treatment with hydrochloric acid, any small amounts of foreign metal initially present may be dissolved out. The temperature of the salt bath and the alkali metal may be maintained at 700–800° C. during the reaction. However, the invention is not restricted to this preferred temperature range. Any alkali metal vapor formed may be condensed, e. g., on the cover of the reaction vessel or by other suitable means and so returned to the reaction zone.

Preferably, the alkali metal is kept in motion during the duration of the reaction, by means of a suitable mechanical stirring device or by a current of inert gas.

The fused salt bath may consist of one or more alkali metal chlorides or mixtures of alkali metal chloride and alkaline earth metal chloride. Fused potassium chloride may advantageously be used alone as the initial bath, since the sodium chloride formed by the reaction will form a low-melting eutectic therewith, whereby the temperature of the bath finally may be held at a relatively low temperature. It should be understood, however, that the invention is not restricted to the above preferred salt bath compositions. Primarily, the invention does not depend on the use of any particular fused bath,

since any fused salt which is substantially chemically inert towards the alkali metal, the titanium salt being reduced and the metal product may be utilized.

The herein described invention is advantageous chiefly in effecting substantially complete utilization of the alkali metal in reducing a volatile titanium compound by reaction with alkali metal. By the present invention, encrustation or coating of the alkali metal with reaction products is effectively prevented, since such products, e. g., NaCl are immediately dissolved by the fused salt bath.

I claim:

1. The process which comprises floating a layer of molten sodium on the surface of a bath of fused potassium chloride and passing the vapor of titanium tetrachloride into said bath beneath said layer of sodium.

2. The process for the production of metallic titanium which comprises floating a layer of molten alkali metal on the surface of a bath of fused potassium chloride at a temperature in the neighborhood of 700–900° C. and passing a volatile titanium compound into said bath beneath said layer of said alkali metal.

3. The process for the production of metallic titanium which comprises floating a layer of molten sodium on the surface of a bath of fused potassium chloride at a temperature in the neighborhood of 700–900° C. and passing a volatile titanium compound into said bath beneath said layer of sodium.

4. A process which comprises reacting a molten alkali metal with a volatile titanium compound to form titanium metal, said molten alkali metal floating on a fused salt bath which is capable of dissolving the alkaline compound formed, is substantially chemically inert to the alkali metal, the titanium compound and metallic titanium, and is composed principally of alkali metal salt.

5. A process according to claim 4 which comprises floating the layer of molten alkali metal on top of the fused salt bath and maintaining said layer of molten alkali metal in motion by stirring during the introduction of said titanium compound.

6. A process according to claim 4 which is operated at a temperature in the neighborhood of 700–900° C.

7. A process according to claim 4, in which the volatile titanium compound is introduced into the fused salt bath below the floating layer of alkali metal.

8. A process which comprises reacting a molten alkali metal with a volatile titanium compound to form titanium metal, said molten alkali metal floating on a fused salt bath which is capable of dissolving the alkaline compound formed, is substantially chemically inert to the alkali metal, the titanium compound and metallic titanium, and is composed principally of alkali metal halide.

9. A process for the production of metallic titanium which comprises floating a layer of molten sodium on the surface of a bath of fused potassium chloride at a temperature in the neighborhood of 700–900° C. and passing titanium tetrachloride into said bath beneath said layer of molten sodium.

10. A process which comprises reacting a molten alkali metal with a volatile titanium compound to form titanium metal, said molten alkali metal floating on a fused salt bath which is capable of dissolving the alkali compound formed, is substantially chemically inert to the alkali metal, the titanium compound and metallic titanium, and is composed principally of alkali metal and alkaline earth metal salts.

11. A process which comprises reacting a molten alkali metal with a volatile titanium compound to form titanium metal, said molten alkali metal floating on a fused salt bath which is capable of dissolving the alkali compound formed, is substantially chemically inert to the alkali metal, the titanium compound and metallic titanium, and is composed principally of alkali metal and alkaline earth metal halides.

12. A process which comprises reacting molten sodium with titanium tetrachloride, said molten sodium floating on a fused salt bath composed principally of sodium chloride and calcium chloride.

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