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PRODUCTION OF METALLIC TITANIUM

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This invention relates to the production of metallic titanium and has for an object the provision of an improved method or process for producing high-purity metallic titanium. More particularly, the invention contemplates the provision of an improved method or process for producing high-purity metallic titanium by dissociation of titanium tetraiodide.

It has been proposed, heretofore, to form high-purity metallic titanium by placing a small amount of iodine and crude titanium metal in a dissociation and deposition vessel having an electric filament of tungsten or other refractory metal disposed therein and capable of being evacuated, and simultaneously heating the vessel and the filament disposed therein. Heating of the vessel causes the iodine and the components of the crude metal to react with the production of titanium tetraiodide. Vapor of the titanium tetraiodide passes into contact with the heated filament where it is dissociated into titanium metal and free iodine. The metal is deposited on the surface of the filament, and the iodine again contacts crude metal, forming additional titanium tetraiodide. The titanium tetraiodide is formed and dissociated with the deposition of titanium until all of the available titanium of the crude titanium metal has been reacted with the iodine or until operation of the apparatus is discontinued for some other reason.

In the operation of the process according to heretofore customary procedures, reaction of the iodine with the metallic titanium under the conditions employed is relatively slow, and the gaseous product of the reaction may contain such contaminants as iodine and products of reaction of the iodine with elements other than titanium present as impurities in the crude titanium-bearing metal. Such contaminants interfere with the deposition of the pure titanium metal on the filament and, also, tend to contaminate the deposited metal.

I have found that the efficiency of the titanium deposition process and the quality of the metallic titanium produced can be improved substantially by employing a preformed gaseous product consisting essentially of titanium tetraiodide instead of employing a mixture of iodine and crude titanium-bearing material in the same vessel as the filament or in a vessel communicating directly with the vessel containing the filament. I have found, also, that the efficiency of the deposition process and the quality of the metallic titanium produced can be improved by maintaining in the dissociation and deposition chamber a pressure of titanium tetraiodide and its dissociation products not higher than that equivalent to about thirty millimeters (30 mm.) of mercury and not lower than that equivalent to one-half millimeter (0.5 mm.) of mercury. I prefer to operate under the higher pressures in the range 0.5 mm. to 30 mm. to secure higher yields of metallic titanium.

According to a method or process of the invention, a gaseous product consisting essentially of titanium tetraiodide is passed in contact with a surface heated to and maintained at a temperature in the range 1100° C. to 1700° C. Contact of the titanium tetraiodide with the heated surface results in dissociation of the titanium tetraiodide with the production of elemental iodine and deposition of metallic titanium of high purity. The deposition surface may be formed of tungsten or titanium or other suitable material, and it may be heated in any suitable manner.

The titanium tetraiodide product employed in carrying out a method or process of the invention preferably

is formed by passing gaseous iodine in contact with any suitable titanium-bearing material capable of reacting with iodine to produce titanium tetraiodide such, for example, as a crude titanium-bearing metal product, at an elevated temperature above the boiling point of titanium tetraiodide (379° C.) and forming a gaseous product containing titanium tetraiodide, iodine and one or more iodides of one or more elements other than titanium if such elements are present in the crude titanium-bearing material and if they are capable of reacting with iodine to form iodides. The gaseous titanium tetraiodide-bearing product is subjected to a fractional cooling treatment to separate various elements or compounds contained therein in accordance with their boiling points and condensation characteristics, or the gaseous titanium tetraiodide-bearing product is cooled to condense all of its various components, and the condensate, thereafter, is fractionally distilled to separate and recover pure titanium tetraiodide. Fractional distillation preferably is carried out at temperatures between the melting point and the boiling point of titanium tetraiodide, in the range 150° C. to 379° C. Any suitable pressure which will permit attainment of the results sought may be employed.

The pure titanium tetraiodide produced by the fractional condensation and fractional distillation methods described is passed in the form of vapor in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. The titanium tetraiodide which contacts the heated surface is dissociated into elemental or metallic titanium and gaseous elemental iodine. The elemental or metallic titanium produced is deposited on the heated surface.

The gaseous elemental iodine produced is utilized in the process for treating additional titanium-bearing material for the production of titanium tetraiodide. The elemental iodine produced by the dissociation reaction may be used directly in the vapor state or after condensation to the liquid or solid state for treating additional titanium-bearing material.

I claim:

1. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, and passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range of about 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect decomposition of the titanium tetraiodide and deposition of metallic titanium on the heated surface.

2. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, and passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect decomposition of the titanium tetraiodide and deposition of metallic titanium on the heated surface.

3. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and

iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, and utilizing the elemental iodine of the gaseous product in the treatment of additional titanium-bearing material.

4. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, and utilizing the elemental iodine of the gaseous product in the treatment of additional titanium-bearing material.

5. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate at a temperature in the range 150° C. to 379° C. to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, and subsequently passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect decomposition of the titanium tetraiodide and deposition of metallic titanium on the heated surface.

6. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate at a temperature in the range 150° C. to 379° C. to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, and subsequently passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect decomposition of the titanium tetraiodide and deposition of metallic titanium on the heated surface.

7. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, and passing the gaseous product containing iodine in contact with titanium-bearing material to form additional titanium tetraiodide.

8. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, and passing the gaseous product containing iodine in contact with titanium-bearing material to form additional titanium tetraiodide.

9. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, cooling and condensing the gaseous product containing iodine, fractionating the condensate thus produced to separate the iodine, and utilizing the separated iodine to form titanium tetraiodide.

10. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling and condensing the gaseous product, fractionally distilling the condensate to produce a gaseous product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure not higher than about 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, cooling and condensing the gaseous product containing iodine, fractionating the condensate thus produced to separate the iodine, and utilizing the separated iodine to form titanium tetraiodide.

11. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and iodides of elements other than titanium, and passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range of about 1100° C. to 1700° C. at a pressure of titanium tetraiodide and its dissociation products in the range 0.5 mm. to 30 mm. to effect decomposition of the titanium tetraiodide and deposition of metallic titanium on the heated surface.

12. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises passing gaseous iodine in contact with the crude titanium-bearing material at an elevated temperature and forming a gaseous product comprising titanium tetraiodide and iodine, cooling the gaseous product and separating iodine and other contaminants therefrom to form a product consisting essentially of titanium tetraiodide and free of iodine and

iodides of elements other than titanium, passing the titanium tetraiodide product in gaseous form in contact with a heated surface maintained at a temperature in the range 1100° C. to 1700° C. at a pressure of titanium tetraiodide and its dissociation products in the range 0.5 mm. to 30 mm. to effect dissociation of the titanium tetraiodide with the production and deposition of metallic titanium on the heated surface and the production of a gaseous product containing elemental iodine, and utilizing the elemental iodine of the gaseous product in the treatment of additional titanium-bearing material.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,497,417	Weber	June 10, 1924
1,671,213	Van Arkel et al.	May 29, 1928
1,891,124	Van Arkel et al.	Dec. 13, 1932
2,393,264	Rentschler et al.	Jan. 22, 1946