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.

According to some heretofore customary procedures, metallic titanium of high purity is produced by contacting titanium tetraiodide with a heated surface 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 iodine and titanium of the titanium tetraiodide with the deposition of the titanium on the heated surface and with the production of a gaseous product containing the iodine in vapor form. Titanium or tungsten or some other suitable metal is employed in forming the heated surface.

The titanium tetraiodide employed is formed by contacting iodine in vapor form with crude metallic titanium. The dissociation operation is carried out in a closed vessel or chamber under high vacuum, and the amount of titanium contained in each unit of volume of the vessel or chamber, and, consequently, the amount of titanium available for deposition from each unit of titanium tetraiodide vapor therein, is relatively small.

The present invention provides a method of increasing the efficiency of the deposition operation by making available for deposition a greater amount of titanium in each unit of volume of the deposition vessel or chamber.

The invention utilizes that property of titanium triiodide which permits it to remain stable at elevated temperatures in the presence of titanium tetraiodide, and, in utilizing that property, the invention employs, in the deposition vessel or chamber a mixture of titanium triiodide and titanium tetraiodide instead of titanium tetraiodide alone.

When the mixture of iodides contacts the heated surface, the tetraiodide is dissociated, disturbing the equilibrium and permitting progressive dissociation of the triiodide in accordance with the following equations:

(1) \[ 2\text{TiI}_4 = \text{TiI}_3 + \text{I}_2 \]
(2) \[ 2\text{TiI}_3 = \text{TiI}_2 + \text{I}_2 \]
(3) \[ \text{TiI}_4 = \text{TiI}_2 + 2\text{I}_2 \]

In a preferred process of the invention, iodine in vapor form is contacted with titanium carbide at an elevated temperature such, for example, as 1100°C, to produce a gaseous product comprising titanium tetraiodide, the resulting gaseous product is contacted with crude titanium metal such as scrap metal at a temperature in the range 600°C to 1000°C, to produce a stable mixture of titanium tetraiodide and titanium triiodide, and the mixture is contacted with a heated surface at a temperature in the range 1100°C to 1700°C, to produce metallic titanium and a gaseous product containing elemental iodine.

The gaseous products containing elemental iodine are utilized in the production of additional titanium iodides. A gaseous product comprising elemental iodine preferably is contacted with titanium carbide at a temperature of about 1100°C, to form a gaseous product containing titanium tetraiodide which is contacted with metallic titanium at a temperature in the range 600°C to 1000°C, to convert a portion of the titanium tetraiodide to titanium triiodide to regenerate the gaseous product containing titanium tetraiodide and titanium triiodide for re-use in the deposition operation.

I claim:

1. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises contacting a gaseous product comprising titanium tetraiodide and titanium triiodide with a heated refractory surface maintained at a temperature in the range 1100°C to 1700°C, to effect dissociation of the titanium tetraiodide and the titanium triiodide with the production and deposition of metallic titanium on the refractory surface and the production of a gaseous product comprising elemental iodine, reacting the elemental iodine thus produced with titanium carbide at a temperature of about 1100°C, to form a titanium product containing titanium tetraiodide, reacting the titanium tetraiodide thus produced with metallic titanium at a temperature in the range 600°C to 1100°C, to convert a portion of the titanium tetraiodide to titanium triiodide, and contacting the regenerative titanium tetraiodide and titanium triiodide in contact with the said refractory surface for the production and deposition of additional metallic titanium.

2. The method of recovering titanium in the form of a high-purity metallic product from crude titanium-bearing material which comprises contacting a gaseous product comprising titanium tetraiodide and titanium triiodide with a heated body of metallic titanium maintained at a temperature in the range 1100°C to 1700°C, to effect dissociation of the titanium tetraiodide and the titanium triiodide with the production and deposition of metallic titanium on the body of metallic titanium and the production of a gaseous product comprising elemental iodine, reacting the elemental iodine thus produced with titanium carbide at a temperature of about 1100°C, to form a gaseous product containing titanium tetraiodide, reacting the titanium tetraiodide thus produced with a crude titanium-bearing material at a temperature in the range 600°C to 1100°C, to convert a portion of the titanium tetraiodide to titanium triiodide, and passing the regenerative titanium tetraiodide and titanium triiodide in contact with the heated body of metallic titanium for the production and deposition of additional quantities of metallic titanium.

References Cited in the file of this patent