This invention relates to a method for producing titanium metal from titanium tetrachloride. More specifically, it relates to a method for producing titanium metal from titanium tetrachloride by reaction with magnesium metal.

Many processes have been proposed for producing titanium metal by employing reducing metals such as magnesium and sodium. Most of these processes, however, form a titanium metal sponge in the presence of the chlorides of the reacting metal to form a solid fused mass. This fused mass in most cases must be chipped out of the furnace to form small lumps which in turn prolonged preliminary treatments to separate the titanium metal from the so-formed metal chloride. The process is involved and costly due to excessive handling. The titanium metal produced by the prior art processes is subjected to contamination upon exposure in transferring the metal from the furnace to the reducing equipment.

An object of this invention, therefore, is to produce titanium metal by reduction of titanium tetrachloride with magnesium metal and removal of the reaction products without exposure of the metal product. A further object is to provide a process which is economical and easy to operate. A further object is to overcome the cumbersome and costly operation of removing titanium metal from a fused magnesium chloride solidified mass. Another object is to remove the reaction products from the titanium metal sponge produced by reduction of titanium tetrachloride with metallic magnesium efficiently and directly. These and other objects will become apparent from the following more complete description of the present invention.

In its broadest aspects this invention contemplates a method for producing titanium metal which comprises reacting titanium tetrachloride and metallic magnesium in a reaction chamber at elevated temperature to produce titanium metal and molten magnesium chloride, simultaneously removing the magnesium chloride from said chamber, and subsequently removing by distillation the residual magnesium chloride and the unreacted metallic magnesium associated with said titanium metal to produce a purified titanium metal product. A feature of the process of this invention is the carrying out of the above steps so that the titanium metal is formed and purified without exposure to air, moisture or leaching liquids.

This invention further contemplates a process for the production of titanium metal in a multi-
out in various types of apparatus. Satisfactory results have been obtained using the apparatus illustrated in Figure 1. It comprises a trizonal apparatus having a reaction zone 11, a condensing zone 12, and a receiving zone 13, the receiving zone communicating with the reaction zone by a reacted aperture 14. The reaction zone of the apparatus is placed in a suitable furnace 15 which is provided with heating means such as gas burners 16.

In carrying out the process of the present invention magnesium metal billets 21 are added through port 20 and fall to the bottom of the reaction zone 11. Titanium tetrachloride vapors 22 are added through port 19 and are reacted with the magnesium metal in the reaction zone. The temperature in the reaction zone is controlled by adjusting the temperature of the furnace 15 which surrounds the reaction zone. Titanium metal and molten magnesium chloride are formed in the reaction zone as reaction products 23. The aperture 14 at the bottom of the reaction zone is of such dimensions as to permit molten magnesium chloride to drain through the aperture, but to prevent titanium metal and magnesium metal from draining through. It has been found that the width of the aperture may vary from 0.001 to 0.125 inches and that the preferred aperture range is from 0.01 to 0.1 inch. The length of the aperture is immaterial. The molten magnesium chloride 24 formed as a reaction product drains through the aperture and is collected in the receiving zone where it solidifies. The residual magnesium chloride which does not drain through the aperture and the unreacted magnesium metal both of which are associated with the titanium metal are distilled and condensed into the condensing zone preferably by reduced pressure thus leaving a purified titanium metal product in the reaction zone. In carrying out the distillation operation means represented as a vacuum pump 17 which communicates with said apparatus by outlet 18, is provided for subjecting the reaction products to a reduced pressure during distillation.

The following example is presented to illustrate a preferred embodiment of the present invention:

**Example 1**

The apparatus shown in Figure 1 was used in this example. 5.0 pounds of magnesium metal were placed in the reaction zone which was heated to 725° C. in furnace 15. Titanium tetrachloride vapors were introduced into the apparatus through port 19 at the rate of 0.5 pound per minute. Additional magnesium metal was added intermittently through port 20 at the rate of one pound every 7½ minutes. The magnesium metal fell to the base of the reaction zone and was reacted upon by the titanium tetrachloride vapors to form titanium metal and molten magnesium chloride. The additions of titanium tetrachloride vapors and the magnesium metal were continued for 2 hours at which time the apparatus reached its capacity. The total amount of magnesium metal added to the furnace was equal to a 20% excess over the amount required to theoretically react with titanium tetrachloride to form titanium metal. This 20% excess insured a substantially complete reduction of titanium tetrachloride to titanium metal. During the reaction the temperature of the reaction zone was held between 900° C. and 980° C. The so-formed molten magnesium chloride was drained through the aperture into the receiving zone where it was solidified. The aperture was positioned at the junction of the side walls and the base of the reaction zone. The aperture extended substantially around the periphery of the base. The width of the aperture was 0.1 inch.

At the end of the reaction most of the magnesium chloride formed had drained through the aperture and away from the reaction zone into the receiving zone. After the reaction was completed all of the titanium metal values along with the residual magnesium chloride and unreacted magnesium metal associated with the titanium metal remained in the reaction zone. The associated magnesium chloride and unreacted magnesium metal were removed from the titanium metal by a distillation operation. The vacuum system including vacuum pump 17 which is connected to the furnace through outlet 18 was employed for the distillation operation. The residual magnesium chloride and unreacted magnesium metal were removed from the titanium metal by distillation in which these substances were maintained at temperatures from 640° C. to 690° C. in the reaction zone and the so-formed vapors were collected as condensate in the cooler condensing zone. The distillation continued for 16 hours. A pressure of less than 10 microns measured by a Pirani gauge placed in the vacuum line outside the furnace was maintained for 3 hours toward the end of the distillation operation. At the end of the distillation operation substantially complete separation of the magnesium metal and magnesium chloride from the titanium metal was obtained. The titanium metal in the furnace was cooled to 25° C. The apparatus was then dismantled and the titanium metal was removed. 15.2 pounds of titanium metal were removed from the reaction zone which represented a recovery of 96%. The titanium metal analyzed above 95% titanium and had a Brinell hardness of 120. The receiving zone contained 55 pounds of magnesium chloride which showed that 87.7% of the magnesium chloride formed was drained through the aperture into the receiving zone. The condensing zone contained 7 pounds magnesium chloride and 4 pounds of magnesium metal which accounted for the residual magnesium chloride and the unreacted magnesium metal which was associated with the titanium metal. The titanium metal produced by this process was ductile and of high purity.

In order to avoid the difficulties involved in assembling and disassembling the apparatus, a preferred type of apparatus is illustrated in Figure 2. In this apparatus the reaction zone 11 is confined in a reaction pot 31; the receiving zone 12 and the condensing zone 13 are combined into one zone which is represented as being confined in the lower portion 32 of the apparatus which is infurposed under the reaction zone.

The operation is carried out in the same manner as previously described. The molten magnesium chloride formed is allowed to drain through the aperture 14 into the receiving zone 12 where it is solidified. The remaining magnesium chloride and unreacted magnesium metal values are removed from the pot in the reaction pot by distillation and are condensed in the condensing zone 13. The vapors are removed from the titanium metal under reduced pressure by vacuum pump 17. The vapors pass over the top of the pot, then downwardly along the outside of the pot through passage-
way 33 and enter the lower portion of the apparatus 32 where they are condensed. Substantially pure titanium metal remains in the reaction pot and is recovered in a more desirable manner than using the other more cumbersome type of apparatus. A multizonal apparatus is necessary in order to remove the molten magnesium chloride away from the titanium metal as it is formed and in order to remove by distillation the remaining magnesium chloride and the unreacted magnesium metal to obtain a substantially pure titanium metal product.

In carrying out the process of this invention, all of the magnesium metal required for the reaction may be placed in the reaction chamber at the onset, but it is more desirable to add the magnesium metal as the reaction proceeds. A more complete reaction apparently is obtained if the magnesium metal is added intermittently. It is also desirable to add the titanium tetrachloride to the reaction chamber in vapor form instead of in liquid form. A method for introducing titanium tetrachloride as a vapor into a reaction chamber is described in patent Application Serial No. 129,308, filed November 25, 1949, now abandoned.

The temperature in the reaction zone should be held above the melting point of MgCl₂, i.e., above 712° C., but below the temperature at which the metal values in the reactor walls are attacked, i.e., above 980° C. the preferred temperature range being from about 720° C. to about 920° C.

The entire operation requires the absence of an oxidizing atmosphere in order to prevent contamination of the titanium metal produced. This may be accomplished in several ways including employment of an inert atmosphere, i.e., helium or argon, or by excluding gases other than titanium tetrachloride from the entire reduction apparatus.

By the process of this invention, removing the magnesium chloride from the reaction zone as the reaction proceeds, it is possible to obtain maximum utilization of the equipment capacity which is not possible in processes which retain all of the magnesium chloride formed in the reaction zone. This provides for greater production of titanium metal per unit volume of equipment capacity.

This invention eliminates the necessity for chipping or boring out a solidified fused mass containing magnesium chloride from the reaction which is required in most of the former processes. This process also eliminates the cumbersome and costly leaching operation and avoids excessive handling. By this process pure titanium metal may be produced by reduction of titanium tetrachloride with magnesium metal and removal of the reaction products without exposure of the metal produced. This process is easy to employ and has decided economical advantages over former processes for producing titanium metal.

While this invention has been described and illustrated by the examples shown, it is not intended to be strictly limited thereto and other modifications and variations may be employed within the scope of the following claims.

We claim:

1. Method for producing titanium metal which comprises forming in a reaction zone a pool of magnesium metal and reacting vaporous titanium tetrachloride with said pool in said reaction zone at elevated temperatures to produce titanium metal and molten magnesium chloride and having present throughout the reaction an excess of magnesium metal over the theoretical amount to react with the titanium tetrachloride and adding thereby maintaining said pool of magnesium metal in said reaction zone, and during said reaction simultaneously, continuously removing magnesium chloride from the titanium metal and the pool of molten magnesium metal in said zone to form easily removable dense titanium metal product.

2. Method for producing titanium metal which comprises forming in a reaction zone a pool of magnesium metal and reacting vaporous titanium tetrachloride with said pool in said reaction zone at elevated temperatures to produce titanium metal and molten magnesium chloride and having present throughout the reaction an excess of magnesium metal over the theoretical amount to react with the titanium tetrachloride, added thereby maintaining said pool of magnesium metal, and during said reaction simultaneously, continuously removing magnesium chloride from the titanium metal and the pool of molten magnesium metal, and continuously removing magnesium chloride from the titanium metal and the pool of molten magnesium metal by distillation in said reaction zone the residual magnesium chloride and the unreacted metallic magnesium associated with said titanium metal to produce an easily removable dense purified titanium metal product.

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