Dec. 17, 1957 M. BENEDICT ET AL 2,816,828

METHOD OF PRODUCING REFRACTORY METALS

Original Filed Aug. 29, 1951 2 Sheets-Sheet 1

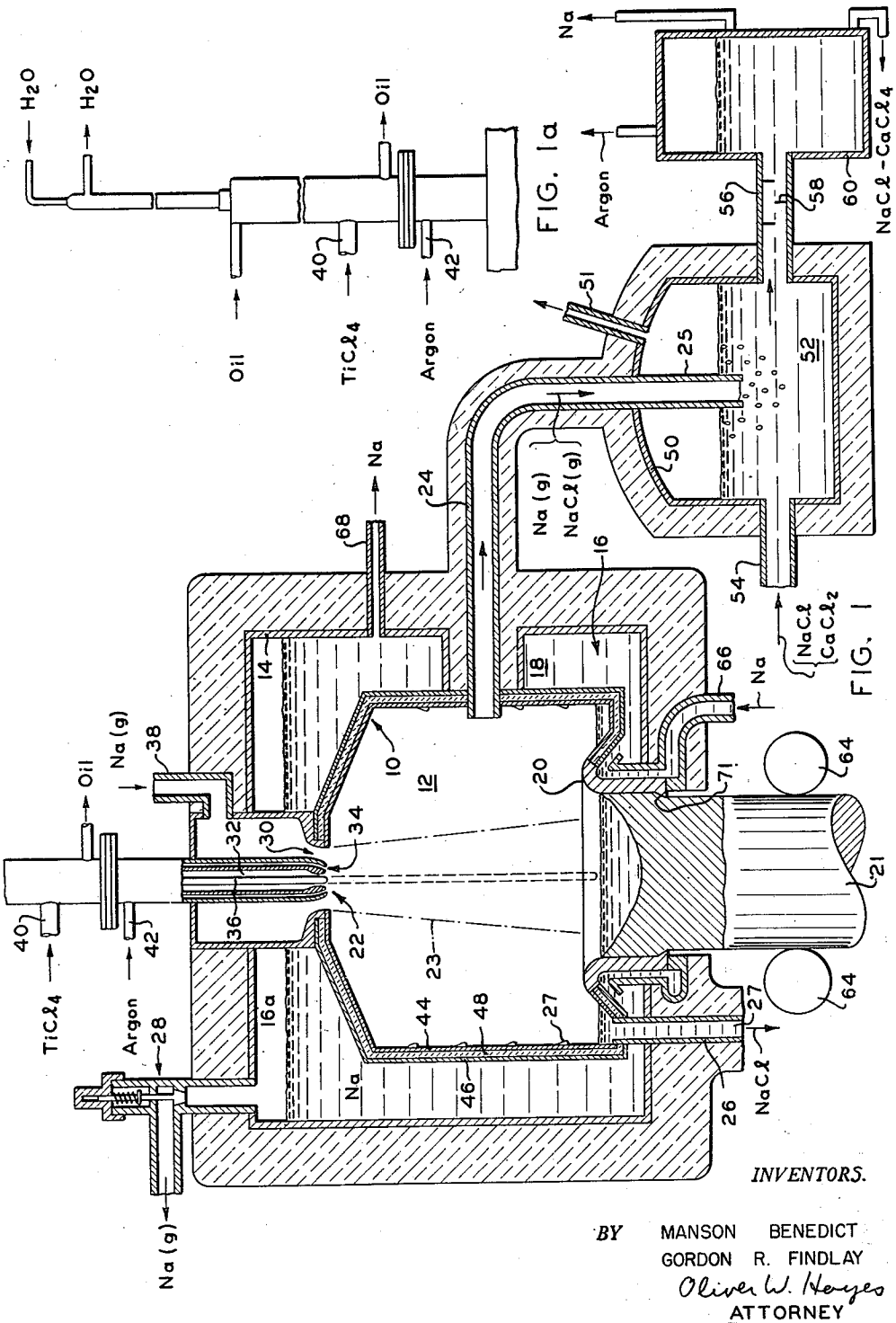


FIG. 1a

FIG. 1

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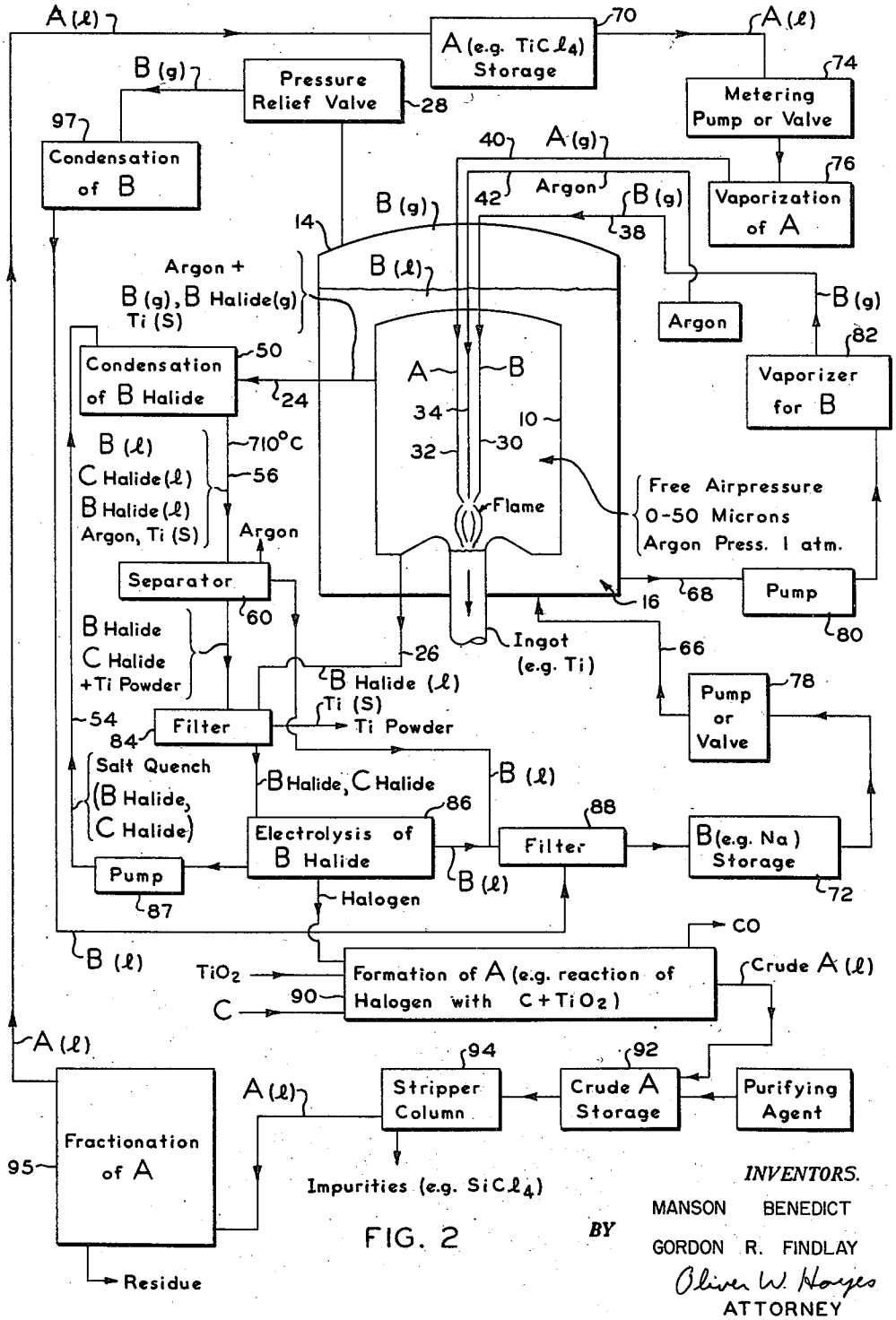


FIG. 2

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United States Patent Office

2,816,828

Patented Dec. 17, 1957

2,816,828

METHOD OF PRODUCING REFRACTORY METALS

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Continuation of application Serial No. 244,138, August 29, 1951. This application June 20, 1956, Serial No. 592,690

8 Claims. (Cl. 75—84.5)

This invention relates to the production of metals and more particularly to the production of metals in a high state of purity. This invention is particularly concerned with improvements in the "metal torch" process of the type described in the application of Gordon R. Findlay, Serial No. 200,606, filed December 13, 1950. This application is a continuation of copending application, Serial No. 244,138, filed August 29, 1951, now abandoned.

A principal object of the present invention is to provide improved processes for the production of metals and alloys thereof, and particularly high-melting-point metals, such as titanium, zirconium, and the like, by the reduction of a halide of such metals.

Another object of the invention is to provide for improved heat dissipation in processes of the above type wherein the by-product halide is rapidly quenched to a temperature where its vapor pressure is extremely low.

Still another object of the invention is to provide an improved apparatus for carrying out processes of the above type, this apparatus limiting the heat flux through the walls of the reaction chamber while permitting extremely high temperatures in the reaction zone.

Still another object of the invention is to provide processes and apparatus of the above type wherein the by-products of the reaction are rapidly condensed in a simplified manner so as to limit the amount of apparatus which is subjected to extremely high temperatures.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the process involving the several steps and the relation and the order of one or more of such steps with respect to each of the others, and the apparatus possessing the construction, combination of elements and arrangement of parts which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawings wherein:

Fig. 1 is a diagrammatic, schematic, sectional view of one preferred modification of the invention;

Fig. 1*a* is a diagrammatic, schematic, sectional view of an extension of the apparatus of Fig. 1; and

Fig. 2 is a diagrammatic flow sheet illustrating the use of the invention.

In general the present invention relates to the production of metals by the reduction of a reducible compound thereof. This reducible compound is preferably one which can be vaporized at a temperature below its decomposition temperature and the invention will, for simplicity of illustration, be initially described in connection with the production of titanium by the reduction of a titanium tetrahalide by an alkali metal or alkali earth metal.

The present invention is primarily directed to improvements in the "metal torch" process and apparatus of the type described in the above mentioned Findlay application. In this "metal torch" titanium tetrachloride, for example, is introduced as a vapor into a reaction zone within a reaction chamber which has an atmosphere inert to titanium. This inert atmosphere is preferably an atmosphere of a gas such as argon. The reducing agent, which is also preferably introduced into the reaction zone in vapor phase is, in a preferred embodiment, an alkali metal such as sodium. The two reactants (i. e., sodium and titanium tetrachloride) are mixed together as they enter the reaction zone where they burn with a highly exothermic reaction to form molten titanium droplets and sodium chloride vapor as by-products of the reaction. The rate of introduction of the two reactants is preferably such that the heat of the reaction is sufficient to melt the product titanium and to maintain the by-product sodium chloride in vapor phase. The reacting gases or vapors are preferably directed towards the molten surface of a titanium ingot so that the product titanium, in liquid form, coalesces on the surface of the ingot. The by-product sodium chloride is preferably separately condensed from the point of collection of the product titanium.

In the above reaction the heat generated is extremely high. The adiabatic flame temperature has been calculated to be on the order of 2800° C. In carrying out such a reaction it is highly desirable that the reaction zone be maintained at the highest possible temperature, at least above the melting point of the product titanium. If possible, it would also be desirable to maintain the walls of the reaction chamber at this high temperature so as to limit radiation heat losses from the flame. However, even when the refractory metals molybdenum or tungsten are utilized for lining the walls of the reaction chamber, it is undesirable to maintain temperatures as high as 1812° C. (M. P. of titanium) at the inner surfaces of these walls, since it is possible for titanium to coalesce on these walls and to form a lower-melting eutectic with the refractory metal of the walls. This is due to the fact that some of the product titanium, even though it is a very small percentage, may not be collected by impingement on the ingot and is free to collect on the walls of the reaction chamber. As a consequence it is desirable to maintain the inner wall of the reaction chamber at a temperature below the melting point of the eutectic formed between the product titanium and the refractory metal of the inner wall. To obviate any possibility of accumulation of titanium particles on the walls of the reaction chamber, the wall temperature is preferably maintained close to, but below the boiling point of, the by-product halide. Thus the by-product halide continuously washes down the surface of the inner wall. When sodium chloride is the by-product, this wall temperature is preferably near, but below, 1465° C.

In the present invention the majority of the by-product halide (i. e., sodium chloride) is condensed in a manner which permits maintaining a high temperature in the reaction zone, while limiting the heat flux through the metal walls of the apparatus. This condensation of the sodium chloride vapors is preferably achieved by quenching these vapors with a liquid which is inert to the sodium chloride. This quenching liquid has a temperature less than the condensation temperature of the sodium chloride, but has a sufficiently high temperature so that the mixture of the liquid and the condensed sodium chloride remains liquid. In one modification of the invention this condensation is achieved outside of the reaction zone and the quenching liquid is preferably a fused salt such as a sodium chloride-calcium chloride mixture. In a preferred embodiment of the invention this sodium chloride-calcium chloride mixture contains about 59% calcium chloride and can be the electrolyte from the electrolysis cell which is utilized for electrolyzing the by-product sodium chloride. This quench liquid can be kept fluid at a temperature sufficiently low so that the

condensed sodium chloride has a very low vapor pressure, thus avoiding any appreciable entrainment of sodium chloride vapors in the re-circulating argon stream.

Referring now more specifically to Figs. 1 and 1*a* there is shown one schematic, diagrammatic illustration of a preferred "metal torch" embodying the present invention. The apparatus comprises a reaction vessel **10** defining therewithin a reaction chamber **12**, this reaction chamber being generally of the type described in the above mentioned Findlay application. Surrounding reaction vessel **10** there is preferably positioned a second vessel **14**, the space **16** between these two vessels being arranged to hold a heat-exchange medium **18**. Near the bottom of the reaction chamber **12** there is located an ingot-forming mold **20** in which a titanium ingot **21** is formed during the reaction.

For obtaining intimate mixture of the titanium tetrachloride and sodium vapors there is provided a "torch" **22** which separately feeds these two vapors into the reaction chamber and directs these two vapors together as they enter the chamber so as to form a flame **23** in which the reduction takes place. This flame **23** serves as the reaction zone and achieves complete reduction of the titanium tetrachloride to metallic titanium. Since the flame **23** is directed towards the ingot mold **20**, the resultant titanium droplets are caused to impinge on the molten surface of the ingot in the mold and to coalesce on this surface, the flame being sufficiently hot to maintain at least the upper surface of the ingot in molten condition.

The reaction chamber **12** includes a vapor line **24** through which the major percentage of the sodium chloride by-product is removed in vapor phase, this vapor line **24** terminating in an extension **25** which leads into a quench chamber **50**, which is more fully described hereinafter. The reaction chamber also preferably includes a vacuum pumping port (not shown) through which a suitable vacuum pumping system can evacuate the reaction chamber to a low free air pressure on the order of less than .001 mm. Hg abs. Located near the bottom of the reaction chamber, and spaced to one side of the ingot mold, is an outlet pipe **26** for removing liquid sodium chloride **27** which condenses on the walls of the reaction chamber **12**.

The vapor pressure of the heat-exchange medium **18** is controlled by a pressure relief valve, generally indicated at **28**, the setting of this pressure relief valve **28** controlling the temperature of the liquid heat-exchange medium **18** as a function of the vapor pressure in the space **16***a* thereabove.

The "torch" **22** comprises, in the preferred form shown, an outer nozzle **30**, through which sodium vapors are adapted to be introduced, an inner nozzle **32**, through which titanium tetrachloride vapors are introduced, and an intermediate nozzle **34**, through which an inert gas such as argon is introduced. The inner nozzle **32** may also include an electrode **36** which, when moved to the dotted line position shown in Fig. 1, may be employed for initially melting the surface of the ingot **21**. The use of such an electrode is more fully described in the copending application of Findlay, Serial No. 245,873, filed September 10, 1951. The details of construction of the "torch" nozzle illustrated in Fig. 1 are described more fully in the copending application of Benedict, Serial No. 244,137, filed August 29, 1951.

The wall **10** defining the reaction chamber is preferably made with a double wall construction, the inner wall **44** comprising a thin refractory metal shell, for example a molybdenum shell, and the outer wall **46** comprising a stainless steel shell. The space between these walls can be filled with a refractory **48**, such as silicon carbide. Equally this space may be filled with an inert gas, heat being transferred between these walls by radiation, as described more fully in the copending application of Findlay, Serial No. 245,873, filed September 10, 1951. Consequently, the major portion of the temperature drop is through the refractory **48** or the inert gas (whichever is used) rather than through the metal of either of the two walls. This temperature drop may be maintained sufficiently high so that the inner refractory metal wall **44** is maintained at a temperature on the order of above 1700° C. in those cases where the possibility of forming a eutectic with the refractory metal is slight. Equally this temperature can be maintained between about 1465° C. (B. P. NaCl) and 1700° C. In this case, none of the sodium chloride condenses in the reaction chamber. It is preferred, however, that the inner wall temperature be maintained at about 1400°–1465° C. With such temperatures only a small percentage of the by-product sodium chloride **27** need be condensed on the inner wall, this condensation being preferably adjusted so that the inner surface of the refractory wall **44** is maintained slightly wet by a film of sodium chloride. Any fine titanium particles which do not coalesce on the surface of the ingot, and which migrate towards the refractory wall **44**, are entrained by the liquid sodium chloride film, running down the inside of this wall, and are washed out of the reaction chamber through the pipe **26** by means of the effluent sodium chloride.

The particles of titanium which are condensed in the reaction chamber as a smoke are carried out through the pipe **24** with the sodium vapors, sodium chloride vapors, and argon passing out of the reaction chamber. The end **25** of the pipe **24** preferably extends below the surface of a quench liquid **52** in a quench chamber **50**. This quench liquid preferably comprises a sodium chloride-calcium chloride mixture containing approximately 59% calcium chloride and may conveniently be the electrolyte from an electrolytic cell utilized for recycling the sodium by electrolyzing the by-product sodium chloride. This sodium chloride-calcium chloride mixture is pumped into the quench chamber **50** through the pipe **54**, the supply of this quench liquid being sufficiently large to completely quench all of the by-product sodium and sodium chloride vapors. The quench liquid may be used in a ratio of about 20 parts, or more, by weight of quench liquid to each part by weight of the vapors to be condensed. The temperature and quantity of the quench liquid are such that the resultant condensed mixture has a temperature near, but above, the freezing point of the mixture formed. The resultant mixture preferably has a temperature sufficiently low (less than 800° C.) so that the sodium chloride has a low vapor pressure, on the order of less than 10^{-3} atm.

When a sodium chloride-calcium chloride mixture is employed as a quench liquid, it preferably has a temperature of about 600° C., being used in sufficient quantities to give a temperature of about 710° C. to the quenched sodium chloride, this being just above the freezing point of the solid solution of sodium and calcium which may be present in the electrolyte used as the quench salt. The outlet pipe **56** through which the condensed mixture is removed from the quench chamber **50** preferably includes baffles **58** which assure thorough mixing of the constituents of the liquid. The mixed liquid then passes into a settling chamber **60** in which any condensed sodium is separated from the salt mixture by stratification and from which the argon may be removed as a gas. Argon may also be continuously or intermittently removed from quench chamber **50** through bleed line **51**.

In a preferred embodiment of the invention the liquid heat-exchange medium **18** is sodium which is introduced into the space **16** by means of a pipe **66**. The sodium preferably enters through pipe **66**, at a lower temperature than the remainder of the sodium **18** in the space **16**, so that this entering sodium removes heat from the walls of the mold **20** at a very high rate, thus maintaining a solid titanium interface at the interior surface of the ingot mold. Liquid sodium may be withdrawn from the space **16** by means of a pipe **68**.

During the reaction the total pressure in the system is maintained at about atmospheric pressure by the argon. The temperature at the inner surface of the reaction chamber **10** is preferably maintained at a relatively high temperature, such as about 1400°–1465° C. This temperature is thus maintained well below the melting point of any eutectic which might form between the product titanium and the refractory metal of the inner wall **44**. This temperature of the inner reaction chamber wall **44** may be readily maintained substantially constant by controlling the temperature of the heat-exchange medium **18** adjacent the outer surface of the outer wall **46**. The temperature of this heat-exchange medium is maintained at a desired constant level by controlling the vapor pressure in the space **16***a* thereabove.

During the build up of metallic titanium in the mold **20**, by the impringement of freshly formed titanium droplets on the surface of the ingot therein, the mold walls are kept below the melting point of titanium by the feed of the heat-exchange medium in contact with the outer surface of these mold walls. The level of the molten titanium in the mold is preferably maintained essentially constant by withdrawing the ingot **21** by means of rolls **64** as titanium is added to the ingot. This rate of withdrawal may be controlled by thermocouples positioned in the mold wall or by other means for indicating the level of the molten titanium in the mold.

While one preferred arrangement for quenching the by-product sodium chloride has been described above, it is intended as being illustrative only and numerous modifications thereof may be made without departing from the scope of the invention. For example the quench liquid may comprise materials other than the electrolyte and may be a liquid metal such as sodium. Equally the quenching may take place partially or wholly within the reaction chamber, this modification being particularly advantageous when the quantity of reactants is sufficiently high so that the temperature of the reaction flame is not unduly lowered by this quenching. In this embodiment the quench liquid may be run down the inner wall of the reaction chamber. This aspect of the invention may be practiced not only with the preferred embodiment wherein an ingot is made in the reaction chamber, but also where the titanium is condensed as a powder and is removed from the reaction chamber as a suspension in the mixture of sodium chloride and calcium chloride resulting from the quenching of the by-product sodium chloride. This slurry of titanium and salt can then be purified by filtration and leaching, the purified titanium powder being then melted in an inert atmosphere to form a consolidated ingot.

The present invention is of wide utility for the manufacture of numerous materials other than titanium, for example, the production of zirconium by the reduction of zirconium tetrachloride with sodium or magnesium. The present invention is particularly applicable to the production of the group IVa and group Va metals (i. e., titanium, zirconium, hafnium, vanadium, columbium and tantalum). These alternative processes are set forth fully in the above-mentioned copending application of Findlay, Serial No. 200,606. As described fully in this co-pending Findlay application, the basic "metal torch" process can be utilized for making a number of pure metals or alloys. Additionally, it can make the product metal in the form of a powder or a partially sintered mass. Equally, while it is preferred that a single reducing agent be used, it is possible, and sometimes desirable, to use a mixture of two reducing agents so that the by-product halide is a mixed halide which, for some purposes, may have a desirably low melting point. The present invention has great utility in all of these modifications of the "metal torch" invention.

Since certain changes may be made in the above process and apparatus without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description, or shown in the accompanying drawings, shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. The process of producing a metal from the class consisting of the metals titanium, zirconium, hafnium, vanadium, columbium and tantalum by reduction of a halide thereof which is volatilizable at a temperature below its decomposition temperature, said process comprising the steps of feeding said metal halide into a reaction zone defined by a metallic wall, providing a reducing metal from the class consisting of the alkali metals and the alkali earth metals magnesium and calcium, feeding said reducing metal into said reaction zone with mixing of said two introduced materials so that they react with intense heat to reduce said metal halide to said metal in a highly heated reaction flame which is at a temperature above the melting point of the product metal and above the vaporization temperature of the by-product halide, the reaction flame being directed against the surface of a body of said product metal so as to maintain said surface at a temperature substantially in excess of the vaporization temperature of the by-product halide essentially by transfer of heat to said surface from the reaction flame, maintaining the metallic wall defining the reaction zone at a temperature below the melting point of the eutectic formed between the product metal and the metal of the wall and above the temperature at which appreciable condensation of the by-product halide occurs so as to prevent undue chilling of the reaction flame, withdrawing from said reaction zone at least a major portion of the vapors of said by-product halide and quenching said withdrawn vapors by mixing together a stream of said vapors and a stream of a liquid which is inert to said by-product halide, said liquid stream prior to mixing with said vapors having a temperature less than the condensation temperature of said by-product halide and said liquid having a sufficiently high temperature so that the mixture of said liquid and said condensed by-product halide remains liquid, the mixture of quench liquid and condensed by-product halide being maintained below the temperature at which the condensed by-product halide has an appreciable vapor pressure.

2. In a process for producing a product metal selected from the group consisting of titanium and zirconium by reduction of a tetrahalide of said metal with a metallic reducing agent selected from the group consisting of the alkali metals and the alkaline earth metals, wherein the vapors of the tetrahalide and vapors of the reducing agent are mixed in a reaction zone defined by a metallic wall so that the vapors react with intense heat to form a highly heated reaction flame which is at a temperature above the melting point of the product metal and above the vaporization temperature of the by-product halide, the reaction flame is directed against the surface of a body of said product metal to maintain said surface molten by transfer of heat from the flame to the surface and to collect on the molten metal surface liquid metal carried in the flame, and heat is removed from the product metal body to solidify the liquid metal at the solid-liquid interface, the improvement which comprises maintaining the metallic wall defining the reaction zone at a temperature below the melting point of the eutectic formed between the product metal and the metal of the wall and above the temperature at which appreciable condensation of the by-product halide occurs so as to prevent undue chilling of the reaction flame, withdrawing the by-product halide vapors from the reaction zone to a quench zone, feeding into the quench zone a quench liquid which is substantially inert to the by-product halide, contacting the by-product halide vapors with the quench liquid to condense the by-product halide vapors in the quench liquid, the weight of quench liquid fed per unit of time to the quench zone being greatly in excess of the weight

A pair of withdrawal rolls 64 is provided for removing the ingot 21 from the reaction chamber as the titanium is added thereto. A reducing die 71 is also preferably provided at the bottom of the ingot mold 20, this die 71 reducing the size of the ingot sufficiently so as to form an essentially smooth surface which forms a seal with die 71 which is substantially vacuum tight. This arrangement prevents escape of vapors from the reaction chamber and also prevents the inleakage of air into the reaction chamber. It is apparent that nitrogen and oxygen must be kept out of the reaction chamber during operation to prevent contamination of the produced metal.

A preferred operation of the device of Fig. 1 and the arrangement of the auxiliary equipment is illustrated best in the flow diagram of Fig. 2 wherein like numbers refer to like elements in Figs. 1 and 1*a*. In this Fig. 2 there is provided a storage chamber 70 for holding the reducible metal compound A (e. g., titanium tetrachloride). A supply tank for holding the metallic reducing agent B (e. g., sodium) is indicated at 72. A pump or valve 74 is included for feeding titanium tetrachloride from the supply 70 to a vaporizer 76 therefor. Titanium tetrachloride vapors (at about 160° C.) pass from vaporizer 76 into pipe 40. A second pump or valve 78 is included for transferring molten sodium from supply 72 to the space 16 surrounding the reaction vessel 10. From space 16 sodium may be transferred by means of a metering pump 80 to a sodium vaporizer 82, the sodium vapors (at about 1000° C.) then passing into pipe 38.

The sodium chloride reaction product in pipe 26 passes through a filter 84 to an electrolysis chamber 86. Any titanium particles which do not collect on the titanium ingot are recovered by the filter 84 and reprocessed to titanium tetrachloride or melted in an arc furnace. The electrolyte from the electrolysis chamber (e. g., sodium chloride and calcium chloride) is pumped by means of the pump 87 through the line 54 into the quench chamber 50, wherein the major proportion of the sodium chloride by-product vapors are condensed. This quench chamber 50 also condenses any unreacted sodium vapors and entrains any titanium smoke which has passed out of the reaction chamber through the pipe 24. From the quench chamber 50 the quench liquid and suspended titanium powders pass to the separator 60 where the sodium is removed by stratification and pumped through a filter 88 to the supply chamber 72. The sodium chloride-calcium chloride mixture passes through the filter 84, where the titanium fines are removed, and then to the electrolysis chamber 86.

The sodium formed in the electrolysis chamber 86 is piped to supply 72 through a filter 88. The chlorine generated in chamber 86 is pumped to a reaction vessel 90 in which titanium tetrachloride is formed by reaction with titanium dioxide and carbon. This manufacture of titanium tetrachloride is well described in chapter 17 of "Titanium, Its Occurrence, Chemistry and Technology," by Barksdale, published (1949) by the Ronald Press Co., New York. The resultant crude titanium tetrachloride is then piped to a crude storage tank 92 at which time a purifying agent such as oleic acid may be added. From the crude storage tank the crude titanium tetrachloride goes to a stripper 94 where some impurities, such as silicon tetrachloride, are removed. It then passes through a fractionation column 95, and the thus purified titanium tetrachloride is then pumped to storage tank 70.

For initially heating the sodium in the space 16 to a high temperature, on the order of 1000° C., there may be provided a separate heater (not shown), the vapors of the sodium condensing at the top of the space 16 and the condensed sodium being recirculated through the heater until the sodium in space 16 has been brought up to the desired temperature. A separate condenser 97 may be provided for condensing excess sodium vapors generated in the space 16 by the heat of the reaction, these vapors being permitted to escape from the space 16 by means of the pressure relief valve, which controls the vapor pressure of the sodium in this chamber and thus controls the temperature of the sodium. The condensed sodium may then be recycled through the filter 88 back to the sodium storage tank 72.

In the operation of the device shown in Figs. 1 and 2 the supply chambers 70 and 72 are filled with titanium tetrachloride and sodium, respectively, some of the sodium being fed to the space 16 so as to fill this space to the level indicated. The sodium in space 16 may then be heated to a relatively high temperature on the order of about 1000° C. During this heatup time the reaction chamber 12 is preferably evacuated to a free air pressure on the order of less than about 1 micron Hg abs. In lieu of evacuating chamber 12 it may be purged of air by sweeping with argon introduced through pipe 42 at a pressure slightly in excess of atmospheric pressure. When most of the air has been removed from the reaction chamber, the electrode 36 may be moved from the full line position of Fig. 1 to the dotted line position and a suitable power supply may be energized so that the upper surface of the ingot 21 in the mold 20 is melted.

When the reaction chamber 12 has been brought up to a desired high temperature, feed of argon down through the pipe 42 is commenced and the electrode 36 is returned to the full line position. The sodium is then pumped into the vaporizer 82 and titanium tetrachloride is pumped into the vaporizer 76. The vapors from these two vaporizers are fed through their respective nozzles into the reaction chamber. As explained previously, the sodium vapors (at about 1000° C.) enter through the outer nozzle 30 while the titanium tetrachloride vapors (at about 160° C.) enter through the inner nozzle 32. A thin blanket of argon passes from the end of nozzle 34 between the titanium tetrachloride and sodium vapors to prevent mixing of these two vapors until they have passed a short distance into the reaction chamber, this feature of the invention being described more fully in the above mentioned Benedict application.

The reactant vapors passing from the "torch" into the reaction chamber ignite with a highly exothermic reaction to give an intensely hot flame 23 in which the sodium completely reduces the titanium tetrachloride to metallic titanium with sodium chloride as a by-product. In order to assure this complete reduction it is preferred that a slight excess of sodium over the stoichiometric quantity be provided in the sodium vapor feed. The reaction flame is directed against the top of the titanium ingot and maintains the upper surface of this ingot in molten condition. The high velocity of the reactant gases, and the consequent high velocity of the liquid titanium droplets formed in the reaction flame, achieves high impingement separation of the titanium droplets by coalescing thereof upon the surface of the molten titanium ingot.

Since the temperature of the flame 23 is extremely high, the by-product sodium chloride remains in vapor phase in the reaction zone and is substantially completely separated from the metallic titanium formed in the flame. A small proportion of the sodium chloride 27 is preferably condensed on the walls of the reaction chamber, this sodium chloride running down these walls and entraining any small particles of titanium which might tend to collect on the walls of the reaction chamber. The majority of the sodium chloride vapors are withdrawn through the pipe 24 where they are quenched in quench chamber 50 by means of the low-temperature sodium chloride-calcium chloride quench liquid which is pumped into this chamber from the electrolysis cell. The withdrawn sodium chloride passes to the electrolysis chamber 86 where it is electrolyzed to sodium and chlorine. The argon passing through the system is separated from the liquid stream either in the quench chamber 50 or in the separator 60 and is recirculated through the system.

of by-product halide vapors fed per unit of time to the quench zone, maintaining the temperature of the quench liquid entering the quench zone at a temperature near but above the temperature at which the liquid contains a solid phase, the mixture of quench liquid and condensed by-product halide being maintained below the temperature at which the condensed by-product halide has an appreciable vapor pressure.

3. The process of claim 2 wherein a minor portion of the vapors of the second metal halide are condensed on the wall of the reaction chamber to maintain a liquid film of condensed second metal halide on said wall, and wherein the major portion of the vapors of the second metal halide are quenched by contacting said vapors with the quenched liquid.

4. The process of claim 2 wherein said quenching step takes place outside of said reaction chamber.

5. The process of claim 2 wherein said quench liquid comprises a calcium chloride-sodium chloride mixture containing about 59% by weight of calcium chloride.

6. The process of claim 2 wherein said second metal comprises sodium and said quench liquid comprises a molten electrolyte from a sodium-electrolysis cell.

7. The process of claim 2 wherein said quench liquid enters the quench chamber at a temperature on the order of about 600° C. and a large excess thereof is used so that the quenched mixture has a temperature of about 700° C.

8. The process of claim 2 wherein said quenching is achieved at a point sufficiently spaced from said reaction zone so that titanium particles in said reaction zone are not chilled below their melting point.

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