Nov. 24, 1964 M. SOCCI 3,158,671

APPARATUS FOR PRODUCING TITANIUM SPONGE

Original Filed July 28, 1955 2 Sheets-Sheet 1

FIG. 2

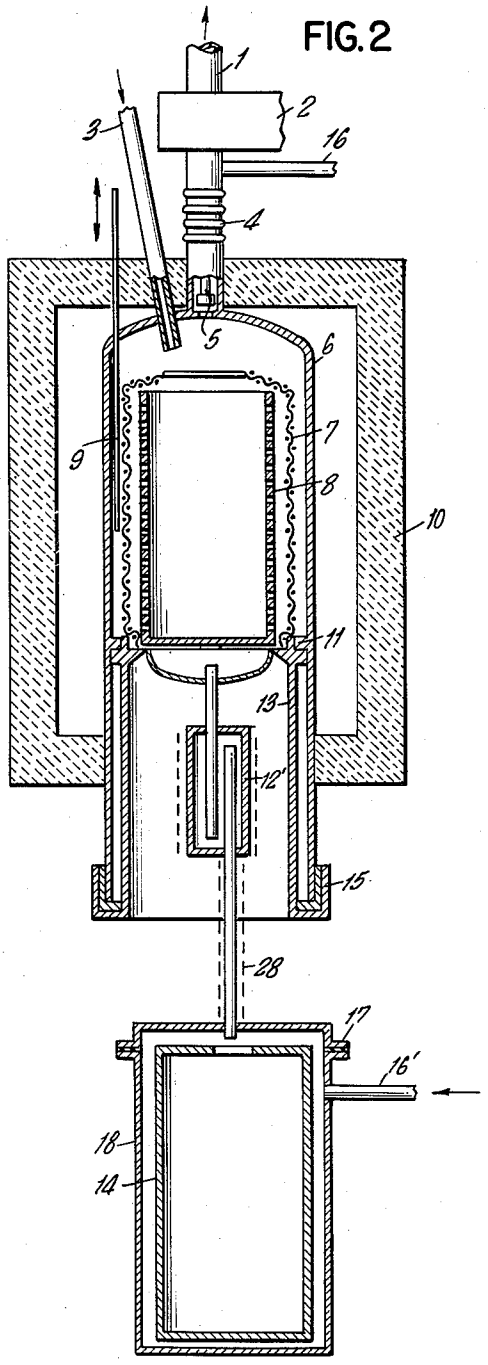
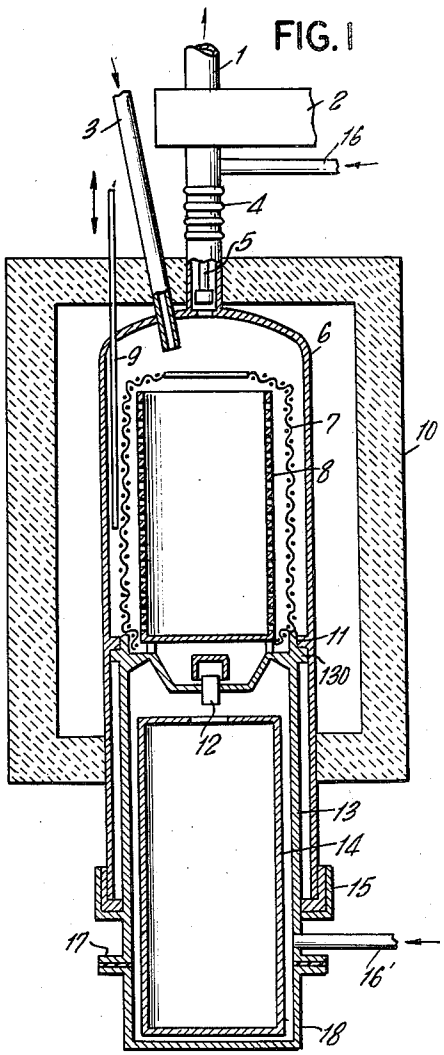



FIG. 1



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APPARATUS FOR PRODUCING TITANIUM SPONGE

Original Filed July 28, 1955 2 Sheets-Sheet 2

FIG.3

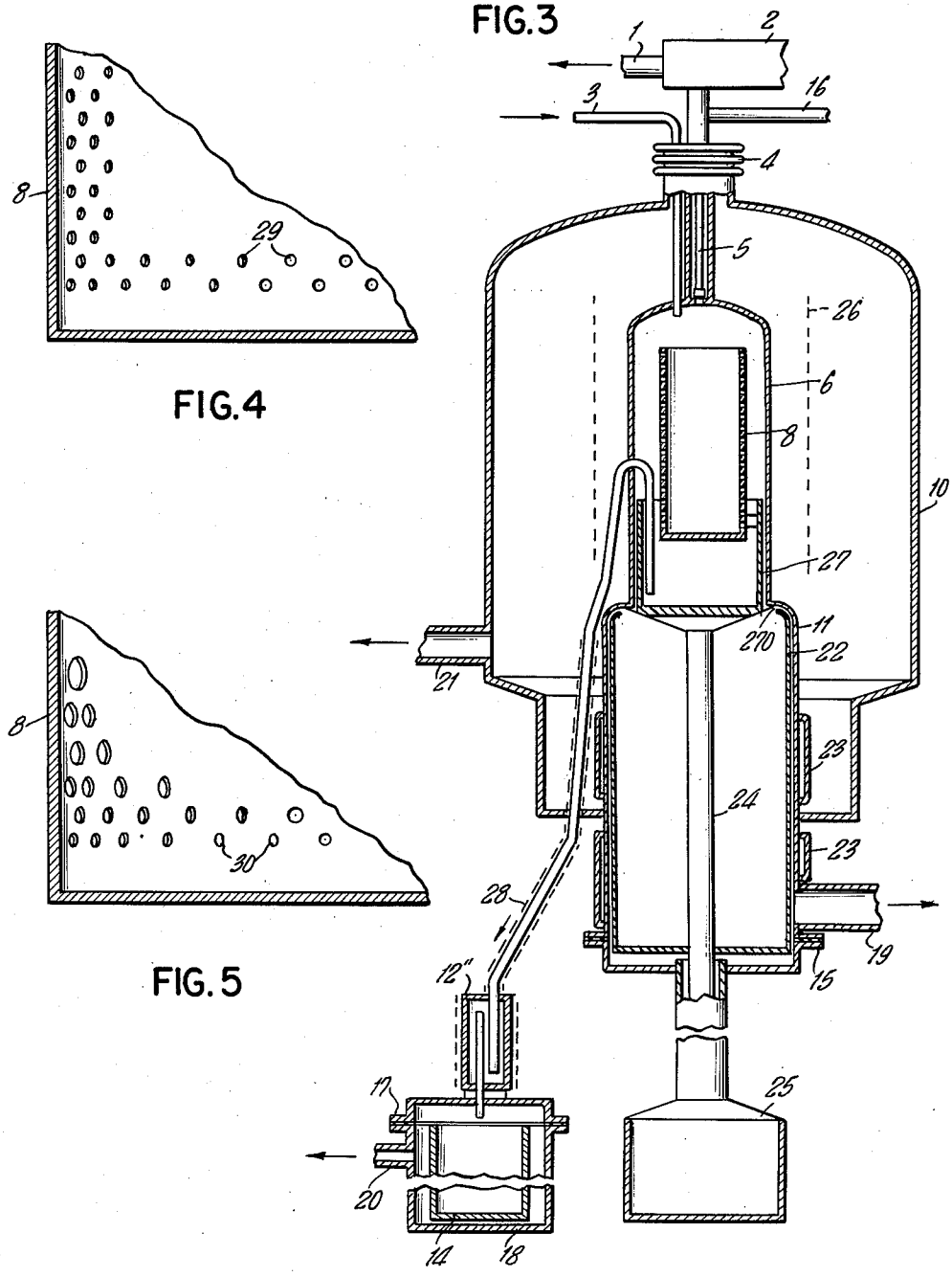






FIG.4




FIG.5

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# 3,158,671
# APPARATUS FOR PRODUCING TITANIUM SPONGE

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Original application July 28, 1955, Ser. No. 525,026. Divided and this application Sept. 9, 1959, Ser. No. 838,948

Claims priority, application Italy Aug. 12, 1954

10 Claims. (Cl. 266—24)

This application is a division of my application Serial No. 525,026, filed July 28, 1955, and now abandoned. The present invention relates to an improved apparatus for producing titanium sponge.

More particularly, this invention relates to an improvement in an apparatus employed for reducing titanium tetrachloride with metallic magnesium according to the equation

$$TiCl_4+2Mg=Ti+2MgCl_2$$

By means of this process, metallic titanium of spongy consistency is produced, a material which is known under the name of titanium sponge. Other reducing substances, such as alkali and alkaline earth metals, can be used instead of magnesium; for reasons of economy, however, the preferred reducing materials are magnesium, sodium, sodium-potassium alloy or mixtures thereof.

The reduction must be carried out under vacuum or in the inert atmosphere of a noble gas, the latter being preferably kept slightly above atmospheric pressure in order to prevent entry of air into the reactor. Titanium reacts readily, even at relatively low temperatures, with both oxygen and nitrogen which, if combined with the metal even in very small amounts, render the latter useless. The reduction itself is a highly exothermic reaction and must be carried out within a temperature range including a lower limit determined by the melting point of the chloride of the reducing metal or of the mixture of the chlorides of the reducing metals, and an upper limit imposed by the boiling point of the reducing metal at the particular operating pressure. Actually, however, a maximum temperature of about 900°–950° C. cannot be exceeded since reaction vessels are commonly made of iron. While at 900°–950° C. the diffusion of titanium and iron into each other is already of considerable magnitude, near 1000° C., the two metals react most violently with each other.

According to prior practice, the reaction is carried out in an iron reactor that can be heated from outside and into which first the reducing metal (for example magnesium in ingots) is charged at room temperature. The reactor is then covered with a gas-tight lid, provided with a vacuum connection, an inert gas inlet and a $TiCl_4$ feeding inlet. The reactor is evacuated in order to remove the air prior to filling it with a noble gas (for example argon). After heating to 750–800° C. and melting the reducing metal, gradual addition of titanium tetrachloride is commenced, whereby the feeding rate is controlled so as not to cause overheating. The formation of titanium sponge starts at the surface of the molten reducing metal, and while the chloride of the reducing metal gathers at the bottom of the reactor, from which it can be periodically drawn off through a drain, a portion of the reducing metal rises by capillary action within the titanium sponge, while the latter continues to increase in height. It is obvious that, under these conditions, the course of the reaction cannot be uniform since, after a period of ready contact between the reactants, the reaction rate passes through a maximum, which must be moderated by cutting the amount of titanium tetrachloride introduced, and then decreases progressively until the reaction practically stops. At this point, about 20–25% of magnesium, which could not react, remain still in the sponge. After cooling the reactor, the titanium sponge formed is removed therefrom by means of a large rake which operates inside the mass but leaves about the reactor wall a thick layer of sponge which is contaminated by the iron of the wall but protects the product obtained in the following operations against such contamination. It is to be noted that the entire operation of opening of the reactor and mechanically removing the sponge must be carried out in an air-conditioned room having a very low relative humidity so as to avoid the absorption of atmospheric moisture by the hygroscopic chlorides which permeate the sponge. Otherwise, the oxygen content of the metal sponge would increase during the subsequent purification by distillation which, obviously, would adversely influence the property of the sponge. It is evident that these precautions add greatly to the production cost.



Another prior method employs a similar reactor, except that a sheet iron container is placed inside thereof so that the bottom of the container, provided with large openings, skims the surface of the molten reducing metal. The formation of the metallic sponge takes place within this container and the sponge is removed from the reactor by lifting out the container. Although this arrangement represents a certain improvement, inasmuch as the use of a rake is thereby avoided, it does not eliminate the principal disadvantages of other prior methods; namely, the difficulty of controlling the course of the reaction and the necessity of repeatedly draining in order to remove the chloride. Aside from being troublesome, this procedure not only causes unfortunately high losses of noble gas but also requires a large excess of reducing metal which, in the case of magnesium, may be as high as 20–25% of the stoichiometric amount. In addition, there is always danger of contamination of the sponge by the walls of the reactor or the container and the consequent necessity of classifying the sponge.

It is the object of the present invention to eliminate these difficulties by providing an improved reactor and feeding it with alternate aliquots of reducing metal and of titanium tetrachloride. To this end, a vessel is placed inside the reactor, which vessel is characterized by having numerous fine perforations at the lateral walls while it has a solid bottom and a wholly or partially open top. By means of suitable feeding devices, the reducing metal and the titanium tetrachloride to be reduced are introduced alternately into this vessel. This design permits the gradual separation of the chloride of the reducing metal from the incipient metallic sponge.

I made the entirely unexpected discovery that if a vessel, provided with suitable lateral perforations as heretofore described, is placed inside the reactor, the molten reducing metal does not pass through the holes, although the chloride formed during the reaction passes therethrough relatively easy. Consequently, the chloride of the reducing metal is continuously separated from the metallic sponge at the very moment of its formation and can be collected in another vessel, also placed inside the reactor, or be continuously withdrawn from the reactor.

Significantly, this percolating of the chloride of the reducing metal through the lateral perforations of the vessel continues even if these perforations have been partially or completely covered by the metallic sponge formed in the course of the reaction.

It is advisable, however, to feed at first only limited aliquots of reducing metal so as to limit the hydrostatic thrust exerted by the column of molten metal onto the

side walls of the vessel, in order to assure that no molten metal will escape through the perforations.

For the above-mentioned reason, it was also found advisable to start the lateral perforations of the vessel at a height of 1 to 3 cm. from the bottom. In this way, the mass of the sponge being formed is isolated from the bottom of the vessel by a thin layer of chloride of the reducing metal, which has the further advantage that where an iron vessel is employed, the removal of its bottom, necessary for the recovery of the sponge, can be carried out without impairing the mass of the metallic sponge.

As the reaction proceeds, the formation of an increasing layer of metallic sponge, which tends to "climb" up the walls of the vessel, causes, as already mentioned, an obstruction of the perforations, rendering them completely impervious to molten reducing metal but not to the chloride formed. Consequently, the aliquots of metal that are added can be gradually increased. It is advisable, however, to always feed the reducing metal in small quantities so that only a thin layer (of the order of 1 to 2 cm.) will be available for the reaction so as to assure that the reducing metal will be always completely used up in the reaction. Generally, the reducing metal may be fed in either the liquid (molten) or solid state; the feeding of the metal in ingots of known weight is preferable, since it permits an exact dosage of the corresponding amount of tetrachloride. Moreover, by introducing cold masses of the metal, some of the reaction heat is removed which contributes toward a more uniform reaction rate. Another reason for feeding the reducing metal in form of solid ingots is that, say, liquid magnesium has a tendency to clog the feeding pipe.

As compared with known devices for this purpose, the herein-disclosed apparatus offers numerous advantages.

(1) It permits the addition of measured aliquots of reducing metal instead of adding it all at once at the start of the operation. As a result, the reaction proceeds at a much more uniform rate, and any dangerous overheatings which, if vessels of iron or iron alloys are employed, may cause alloying with titanium can be readily avoided.

(2) The addition of the charge in small portions permits to substantially avoid the presence of excessive amounts of molten reducing metal, which in the known processes may exceed the theoretical amount by as much as 25%. According to prior processes, such an excess is necessary to avoid the formation of titanium subchlorides as well as because of the fact that according to these processes part of the reducing metal remains included in the metallic sponge formed and thus fails to participate in the reaction.

With the devices and method of operation according to the present invention, this excess can be reduced to 5% and less because any new thin layer of reducing metal resulting from the successive addition of the latter participates in its entirety in the reaction.

(3) Because of what has been said in (2), it is possible to produce, if desired, a sponge mass of metal of considerable height and bulk, by using a vessel of adequate size. This is not possible by means of hitherto known methods and equipment since, without a system of continuously eliminating the chloride of the reducing metal, the metallic sponge retards the reaction to a prohibitive degree upon reaching a certain size, because the reactants do not diffuse with sufficient speed through the spongy mass if the latter is too large.

This, however, does not happen when employing the method and devices of the present invention because the chlorides produced flow continuously from the lateral perforations of the vessel and, because of the feeding system, the course of the reaction is substantially independent from the rate of diffusion of the reactants through the sponge.

(4) While hitherto it was indispensable to increase the diameter of the reactor in order to obtain large masses of metallic sponge, which made it more difficult to remove reaction heat and made it necessary to slow down the reaction, according to the present invention it is possible to obtain large spongy masses in a reactor of relatively small diameter, because the method favors the removal of reaction heat.

(5) Inasmuch as the reaction vessel does not need to function as a mechanical support for the sponge since the growing sponge column supports itself, it can be made from relatively thin iron sheeting or of a suitable alloy; this has the advantage that, at the end of the reaction and after a possible purification by distillation of the metal produced, the body of titanium sponge can be very easily "peeled" by sawing out the bottom of the vessel and making a cut along one of the structural lines thereof.

(6) Referring to what has been said under (5), according to a variation of the present invention, the vessel can be made of a very thin sheet of titanium or any other metal of which a sponge is to be produced. This eliminates any possibility of contamination of the sponge, including damages of the sponge due to accidental overheating. According to this modification, the top limit of the permissible reaction temperature is substantially higher. Moreover, since a body of titanium sponge is obtained that is entirely uniform, no "peeling" or subsequent classification of the sponge is required.

(7) Because of the lateral perforations of the vessel, the final distillation for the removal of the residual chloride of the reducing metal and of any excess of reducing metal can be carried out directly with the spongy mass still enclosed in the vessel. This is of considerable importance, since it permits a quick transfer of the product from the reactor to the still. Thus, any chance of moisture absorption which would impair the characteristics of the product through oxidation, etc. is practically eliminated. Moreover, the reactor itself can be readily adapted for this distillation and the vessel does not need to be taken out at all.

The novel features which I consider characteristic of my invention are set forth with particularity in the appended claims. The invention itself, however, and any additional objects and advantages thereof will best be understood from the following description of several preferred embodiments when read in conjunction with the accompanying drawings, in which—

FIG. 1 represents a vertical section of one embodiment of a reactor according to the present invention;

FIG. 2 represents a vertical section of another embodiment of such a reactor;

FIG. 3 represents a vertical section of a third embodiment of a reactor according to this invention;

FIG. 4 represents a detail of one embodiment of perforated reaction vessel according to the invention, and

FIG. 5 represents a detail of another embodiment of perforated reaction vessel according to the invention.

Similar or corresponding parts of the various embodiments have the same reference numerals.

Referring in more detail to the drawings, FIG. 1 illustrates a reactor the fixed part of which is substantially composed of a shell 6, preferably made from stainless steel. This shell is open at the bottom and comprises relatively thin walls to facilitate both heating and removal of the reaction heat. A tube 1, fitted with an expansion joint 4, extends from an opening machined into the dome of the reactor shell 6. Through this tube passes the stem of a valve 5 (controlled from the outside), adapted to close this opening, in order to prevent the escape of reaction vapors which otherwise would condense on the cold walls of the tube. Tube 1 is provided for the introduction of ingots of reducing metal (for example magnesium) from a distributing feeder 2. Moreover, the reactor is evacuated and subsequently charged with an inert gas (for example argon) by means of tube 1, the inert gas entering through

the duct 16. One or more feeding tubes 3 enter the dome of the reactor shell for the introduction of titanium tetrachloride. The movable part of the reactor comprises a bottom 18 which supports a drum 14 designed to collect the chloride of the reducing metal. By means of a flange 17, having a rubber packing, the bottom 18 is connected to a cylinder 13 which fits into the shell 6 and may be provided with raising and lowering means not shown in the drawing.

In operating position, the cylinder 13 rests with its top rim 130 against an annular stop 11 of the shell 6 so as to provide a tight closure, if necessary augmented by an asbestos gasket (not shown). The cylinder 13 is fitted with an external annular cup 15 which can be heated (by heating means not shown in the drawing). Upon sufficiently raising the cylinder 13, the free bottom rim of shell 6 fits into this cup which is designed to contain sealing lead that is melted each time before the parts 6 and 13 are to be connected or disconnected (that is, before raising or lowering of part 13). Extending laterally from the cylinder 13 is a duct 16', to provide an additional outlet and inlet, respectively, for the vacuum and for the introduction of inert gas in conjunction with duct 16 of tube 1.

The top of cylinder 13 is provided with a cup-shaped cover, fitted with a center siphon overflow 12 for the purpose of continuously discharging the chloride of the reducing metal into drum 14 and acting at the same time, by virtue of the molten salt collecting there, as a hydraulic seal to prevent the escape of vapors present in the reaction zone and their condensation at the cold parts of the bottom. The action of the seal 12 is governed by a pressure equalizer (not shown) which controls the respective rate of flow of noble gas into the top and bottom section of the reactor through ducts 16, 16'.

The lower member 13 supports a reaction vessel 8 which has a solid bottom plate but, from a height of about 1 to 3 cm. upwards, is provided with a dense pattern of minute perforations which, if magnesium is used as a reducing metal, must have a diameter between 0.5 and 3 mm., preferably between 1 and 2 mm. The perforations can be either all of the same size (29, FIG. 4) or their size may increase from the bottom towards the top (30, FIG. 5). The distance between the individual perforations will suitably vary from half to twice their diameter. The internal walls of reactor shell 6 can be advantageously protected against incrustations by the product or by-products by means of a screen 7 inserted between the shell 6 and the reaction vessel 8. If a screen 7 is provided, it may be supported, as shown, by the upper rim of cylinder 13. The vessel 8 is similarly supported, but care must be taken that molten salt, flowing from the lateral perforations of the reaction vessel 8, can easily reach the siphon cup 12. The thermocouple 9 inserted between reactor wall and screen can be shifted vertically up and down so as to allow to regulate the course of the reaction by following the growth in height of the body of metallic sponge which is formed in vessel 8 and to adjust the feeding rate of tetrachloride according to the temperature at the reaction zone. The heating of the top section of the reactor, including the zone of valve 5 and of cup 12, is accomplished by means of a furnace 10 enclosing the reactor. This furnace can be a gas, oil or electric furnace which, however, must be easy to cool. One way of attaining quick cooling is by means of radial jets of cold air entering the interior of the furnace and distributed about the reaction zone at various heights, in order to both moderate the temperature during the reaction and to quickly cool the device at the end of the reaction.

The process is started with a cold furnace. To assemble the reactor, drum 14 is placed into the bottom 18, cylinder 13 is joined to part 18 by tightening flange 17, vessel 8 and screen 7 are placed on top of the cylinder, the



lead in cup 15 is melted, and the lower elements of the unit are lifted until they contact stop ring 11.

After solidfication of the lead in cup 15, reducing metal is charged into the feeding distributor 2 and, feeders 2 and 3 being closed against the outside, the unit is first evacuated by means of pipe 16, 16' and then filled through pipes 16, 16' with a noble gas until a slight over-pressure (several cm. Hg) is attained. Thereupon, furnace 10 is heated to the desired temperature, and an alternate feeding is commenced of reducing metal and of the corresponding quantity of tetrachloride, while the course of the reaction is checked by means of themocouple 9. At the end of the reaction and after the device has been completely cooled (which, as stated previously, may be accelerated by means of jets of cold air), the movable parts of the reactor are disconnected after melting the lead, and the unit is disassembled in reversing the steps of the previously-described assembly operation.

The embodiment of FIG. 2 shows a somewhat different cylinder 13 and siphon overflow 12, the latter being of a length (about 30 to 50 cm. are sufficient) slightly in excess of that required for the discharge of molten salts. While the molten salts are kept at melting temperature by heater 28, the purpose of the longer siphon overflow is to maintain inside the reactor a slight overpressure of noble gas and prevent the ingress of air into the device in case the pressure inside the reactor drops below atmospheric pressure. This arrangement permits the discharge of magnesium chloride either into a drum similar to that in FIG. 1 but placed outside the reactor unit or into any open collecting receptacle. In the latter case, the necessary vacuum seal is provided by solidified salt residues from the preceding operation in siphon 12, instead of by flange 17 as in FIG. 1.

In the embodiment of FIG. 3, the reducing section is exactly the same as that of the preceding devices. However, according to this specific design, the vessel 8 is partly inserted into a tank 27, provided to collect molten chlorides. These latter are periodically discharged, through siphon 12 heated by resistor 28, into receptacle 14 enclosed in an air-tight shell 17, 18. To prime the siphon a slight vacuum is produced in the shell by means of an outlet 20; during the intervals between successive siphoning operations, element 12 prevents return of air. The assembly 8, 27 rests on a top plate 271 of a raising and lowering column 24, provided with control means enclosed in a gas-proof box 25. When raised into operating position, the rim 270 of this plate furnishes an hermetic seal for the heated section of the reactor. Flange 15 serves to hermetically join the movable section 24, 27 and 8 to the fixed section 6, 10. The part of shell 6 which is below seal 11 is somewhat enlarged. Placed inside the enlarged part is a condenser 22 consisting, for example, of a sheet iron cylinder, partly surrounded by coolers 23. By means of an outlet 19, the interior is connected with a high power vacuum pumping unit. The furnace 10 is a resistance furnace provided with resistors 26. The furnace mantle is joined in such a manner with shell 6 of the reactor that, by means of nipple 21, a vacuum may be produced inside the furnace. The movable assembly 24, 27 and 8 is controlled by, say, a hydraulic lift not shown in the drawing. The operation is entirely similar to that of the preceding devices, except that here the distillation of the product is carried out within the furnace proper.

After the reaction is completed and the salt melt is siphoned off, vacuum is produced simultaneously in the reactor and in the furnace in order to distill off the residues of salt and any reducing metal contained in the sponge. For this operation, by lowering column 24 for several cm., an annular passage is created through which the vapors of distillation products pass into the bottom section where they condense in condenser 22. As the end of the distillation, column 24 is lowered completely and the purified, spongy mass is thereby transferred into that section of the

reactor from which it can be readily taken out after cooling.

Obviously, numerous variations are possible within the scope of the present invention. Thus, the perforated vessel may vary in shape; it may have different cross sections (circular, polygonal, etc.) or may have the form of a truncated cone. Moreover, the vessel may contain one or more inner vents provided with lateral perforations.

The process and devices herein described have been designed, as previously stated, for the production of titanium sponge, but it is to be understood that other similar metallurgical operations, such as for example the making of a zirconium sponge, may be carried out in this manner. Similarly, chlorides other than tetrachloride may be used as starting materials without departing from the scope of the present invention.

I claim:

1. An apparatus for making titanium sponge by reducing titanium tetrachloride with a reducing metal, comprising enclosure structure means for applying heat, reactor casing means at least partly within said enclosure means, the casing means providing a reaction chamber, a reaction vessel in said reactor casing means, the reaction vessel having a bottom wall sufficiently imperforate to retain thereon a growing body of the resulting titanium sponge and to retain the molten reducing metal and the resulting molten chloride of said reducing metal, the reaction vessel having foraminous side wall surface, the foramina thereof being distributed over said surface circumferentially at a plurality of levels to provide a multiplicity of vents, the foramina being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, the foramina starting at a region sufficiently removed from the bottom wall to retain thereon at least a thin layer of the said resulting chloride, the foramina remaining open throughout the operation, for passage of said chloride, an upper part of the reaction vessel having an opening for ingress of titanium chloride and said reducing metal, conduit means for supplying titanium chloride and said reducing metal to said opening, movable means providing a chamber to receive the molten chloride from the reaction vessel, means for removing the molten chloride from the latter chamber, a condenser, cooling means for the wall surface of the condenser, the movable means and the casing means having cooperating structure for sealing the lower part of the reaction chamber provided by the reactor casing means, means to raise and to lower said movable means, to seal the said cooperating structure upon raising, and to unseal it upon lowering to provide a vent for vapors into the condenser, the vapors constituting residues of salt and reducing metal distilled from the reactor casing means, said condenser being situate in a lower extension of said reactor casing means.

2. An apparatus for making titanium sponge by reducing titanium tetrachloride with a reducing metal, comprising enclosure structure means for applying heat, reactor casing means at least partly within said enclosure means, the reactor casing means providing a reaction chamber, a reaction vessel in said reactor casing means, the reaction vessel having a bottom wall which is imperforate, so as to retain thereon a growing body of the resulting titanium sponge and to retain the molten reducing metal and the resulting molten chloride of said reducing metal, the reaction vessel having a vent in its side wall surface, the vent being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, an upper part of the reaction vessel having an opening for ingress of titanium chloride and said reducing metal, conduit means for supplying titanium chloride and said reducing metal to said opening, movable means providing a chamber to receive the molten chloride from the reaction vessel, means for removing the molten chloride from the chamber, a condenser, cooling means for the wall surface of the condenser, the movable means and the casing means having cooperating structure for sealing the lower part of the reaction chamber provided by the reactor casing means, means to raise and to lower said movable means, to seal the said cooperating structure upon raising, and to unseal it upon lowering to provide a vent for vapors into the condenser, said vapors constituting residues of salt and reducing metal distilled from the reactor casing means, said condenser being situated in a lower extension of said reactor casing means.

3. An apparatus for making titanium sponge by reducing titanium tetrachloride with a reducing metal, comprising enclosure structure means for applying heat, reactor casing means at least partly within said enclosure means, the casing means providing a reaction chamber, a reaction vessel in said reactor casing means, the reaction vessel having an imperforate bottom wall to retain thereon a growing body of the resulting titanium sponge and to retain the molten reducing metal and the resulting molten chloride of said reducing metal, the reaction vessel having foraminous side wall surface, the foramina thereof being distributed over said surface circumferentially at a plurality of levels to provide a multiplicity of vents, the foramina all being open and of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, an upper part of the reaction vessel having an opening for ingress of titanium chloride and said reducing metal, conduit means for supplying titanium chloride and said reducing metal to said opening, movable means providing a chamber to receive the molten chloride from the reactor casing means, liquid-sealed means for removing the molten chloride from the chamber, a condenser, cooling means for the condenser wall, the movable means and the casing means having cooperating structure for sealing the reaction chamber provided by the reactor casing means, means to raise and to lower said movable means to seal the said cooperating structure upon raising, and to unseal it upon lowering to provide a vent for vapors into the condenser, said vapors constituting residues of salt and reducing metal distilled from the reactor casing means, said condenser being situate in a lower extension of said reactor casing means.

4. A batch apparatus for making titanium sponge by reducing titanium tetrachloride with magnesium metal in which chloride of magnesium is continuously separated from the titanium sponge as it is being formed, comprising a reactor shell, a reaction vessel in and spaced from said reactor shell, the reaction vessel having a bottom wall which is imperforate so as to retain a growing body of the resulting titanium sponge and to retain the molten magnesium and the resulting molten magnesium chloride, the reaction vessel having foraminous side wall surface, the foramina thereof being distributed on said surface circumferentially over at least the major part of the circumference, at a plurality of levels, to provide a multiplicity of vents which remain open and operative to remove magnesium chloride, the vents being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, the vents starting at a region sufficiently removed from the bottom wall to retain therein at least a thin layer of the said resulting chloride, the vents starting about 1 to 3 centimeters from the bottom of said vessel and being from 0.5 to 3 mm. in largest dimension, the vents being spaced from each other for a distance from one half to twice their diameter, an upper part of the reaction vessel having provision for ingress of titanium chloride and said reducing metal, a movable casing within the reactor shell, the reactor vessel being mounted on the upper part of the casing, said casing and reactor having cooperating sealing structures for mutual sealing in an air-tight manner, the casing providing a chamber for catch-

ing magnesium chloride draining from the reaction vessel.

5. A batch apparatus for making titanium sponge by reducing a titanium halide with a reducing metal in which a halide of the reducing metal is continuously separated from the titanium sponge as it is being formed, comprising a reactor shell, a reaction vessel in and spaced from said reactor shell, the reaction vessel having a bottom wall which is imperforate so as to retain a growing body of the resulting titanium sponge and to retain the molten reducing metal and the resulting molten halide of said reducing metal, the reaction vessel having foraminous side wall surface, the foramina thereof being distributed on said surface circumferentially over at least the major part of the circumference, at a plurality of levels, to provide a multiplicity of vents which remain open, so that they are operative as long as there is halide of the reducing metal at their levels, the vents having a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten halide, the vents starting at a region sufficiently removed from the bottom wall to retain therein at least a thin layer of the said resulting halide, the vents starting about 1 to 3 centimeters from the bottom of said vessel and being from 0.5 to 3 mm. in largest dimension, the vents being spaced from each other for a distance from one half to twice their diameter, an upper part of the reaction vessel having provision for ingress of titanium halide and said reducing metal.

6. A batch apparatus for making a refractory metal by reducing a volatile halide thereof with a reducing metal, in which the halide of the reducing metal is continuously separated from a body of the metal as the latter halide is being formed, comprising a reactor shell, a reaction vessel in and spaced from said reactor shell, the reaction vessel having a bottom wall which is imperforate so as to retain a growing body of the resulting refractory metal and to retain the molten reducing metal and the resulting molten halide of said reducing metal, the reactor vessel having foraminous side wall surface, the foramina thereof being distributed on said surface circumferentially over at least the major part of the circumference, at a plurality of levels, to provide a multiplicity of vents which remain open, so that they are operative as long as there occurs metal halide formation at their levels, the vents being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, the vents starting at a region sufficiently removed from the bottom wall to retain therein at least a thin layer of the said resulting chloride, the vents starting about 1 to 3 centimeters from the bottom of said vessel and being from 0.5 to 3 mm. in largest dimension, the vents being spaced from each other for a distance from one half to twice their diameter, an upper part of the reaction vessel having provision for ingress of the halide of the refractory metal and said reducing metal.

7. A batch apparatus for making titanium sponge by reducing a titanium halide with a reducing metal in which the halide of the reducing metal is continuously separated from the titanium sponge as said halide is being formed, comprising a reactor shell, a reaction vessel in and spaced from said reactor shell, the reaction vessel



having a bottom wall which is imperforate so as to retain a growing body of the resulting titanium sponge, the reactor vessel having foraminous side wall surface, the foramina thereof being distributed over said surface circumferentially over at least the major part of the circumference, at a plurality of levels, to provide a multiplicity of vents, the vents being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, the vents remaining open and operative to remove the halide of the reducing metal, an upper part of the reaction vessel having provision for ingress of titanium halide and said reducing metal, means for supplying titanium halide and said reducing metal.

8. A batch apparatus for making refractory metal by reducing a volatile halide thereof with a reducing metal, in which the formed halide of the reducing metal is continuously separated from a body of the refractory metal as the halide is being formed, comprising a reactor shell having a lower opening, a reaction vessel in and spaced from said reaction shell, the reaction vessel having a bottom wall which is imperforate so as to retain a growing body of the resulting titanium sponge, the reaction vessel having foraminous side wall surface, the foramina thereof being distributed over said surface circumferentially over at least the major part of the circumference, at a plurality of levels, to provide a multiplicity of vents, the vents being of a diameter sufficiently small to retain the molten reducing metal but large enough to pass said resulting molten chloride, the vents remaining open and operative to remove reducing metal halide at their levels, an upper part of the reaction vessel having provision for ingress of titanium halide and said reducing metal, means for supplying halide of the refractory metal, and said reducing metal, a movable casing within the reactor shell, the reaction vessel being mounted upon the casing and being removable with said casing from said reactor shell through the lower opening of the latter, said casing and reactor having cooperating sealing flanges for mutual sealing in a gas-tight manner, said casing having a chamber for receiving the molten chloride, and a liquid-sealed outlet for said molten chloride from said chamber.

9. The apparatus defined in claim 8, the foramina being distributed over substantially the entire circumference and over at least the major part of the height of said reaction vessel.

10. The apparatus defined in claim 4, the foramina being distributed over substantially the entire circumference and over at least the major part of the height of said reaction vessel.


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