APPARATUS FOR PRODUCING METAL SPONGES, INCLUDING CONDUIT SEALING MEANS


Application April 15, 1953, Serial No. 349,043

2 Claims. (Cl. 266—24)

This invention relates to an apparatus for producing sponges of refractory metals, such as titanium and zirconium. More particularly, it relates to an improved apparatus in which the process for producing such sponges by reacting a molten alkali or alkali earth metal with a halide of the refractory metal may be carried out.

Such a process is disclosed and claimed in a copending application, Serial No. 353,322, filed May 6, 1953, in the names of Nicholas A. Ziegler and John Wulff. Apparatus over which the apparatus claimed herein is an improvement is likewise disclosed and claimed in copending applications, Serial Nos. 349,042 and 349,041, both filed April 15, 1953, in the names of Mitchell J. Fleiszar, James R. Goldsmith, John F. Lenc and John J. Pilkunus, this present application being a continuation-in-part of these last two previously filed applications.

In the process and apparatus disclosed in these three copending applications, the molten alkali or alkali earth metal and the halide of the refractory metal are simultaneously fed into a suitable reaction chamber maintained preferably at a temperature of about 1500°F. to form a sponge of refractory metal of unusually great density at an unusually rapid rate.

It is an object of this present invention to provide an apparatus which permits the introduction of the two reactants, namely the alkali or alkali earth metal and the halide of the refractory metal, in closely controlled amounts so that better control over the rate of the reaction is obtained. Likewise, it is also an object to permit the introduction of such reagents in approximately stoichiometric amounts, and at a rate which will maintain the evolution of heat from the exothermic reaction within desirable limits.

Still another object of this invention is to permit the introduction of the molten alkali or alkali earth metal in such a way that it is allowed to fall freely from a point within the conduit leading to said reaction vessel, through the height of that reaction vessel. Thus any oxide film which might envelope the individual drops or stream of molten metal is immediately broken at the bottom of this free fall.

Another object is to provide an apparatus of the characteristic which permits the ready starting and stopping of the flow of refractory metal by the action of a control mechanism.

Yet another object of this invention is to provide an apparatus in which the initially formed sponge of refractory metal is continually impregnated with additional reactants during the course of the reaction so that the initially-formed sponge of relatively low density gradually assumes a higher density as the reaction proceeds.

Another object of this invention is to provide an apparatus which will speed up the reaction process by permitting more rapid cooling of the reaction product than has previously been possible.

Another object of this invention is to provide an apparatus which will prevent the reaction product from becoming contaminated by the atmosphere, and which will also reduce the possibility of accidental combustion of the reaction product.

Further objects and advantages of this invention will become evident as the description proceeds and from an examination of the accompanying drawing which illustrates one embodiment of the invention and in which similar numerals refer to similar parts throughout the several views.

In the drawing:
Figure 1 is a somewhat diagrammatic perspective view of one form of apparatus embodying the invention, only fragmentary portions of some of the apparatus being shown;
Figure 2 is an enlarged view in side elevation and partly in vertical cross section of a portion of the conduit connecting the metal melting unit to the reaction chamber, shown in Figure 1;
Figure 3 is an enlarged view in vertical cross section of a portion of the conduit shown in Figure 2;
Figure 4 is an enlarged sectional view of a portion of the apparatus shown in Figure 2, the view being taken along the line 4—4 of that figure.

Referring now to Figure 1, one form of apparatus embodying the invention is illustrated therein. A reaction chamber 10 may be provided having any suitable construction, such as that shown in the two copending applications mentioned above, for example. The upper end of the reaction chamber may be provided with a suitable cover 12 which is preferably disposed in sealed relation to the reaction chamber 10 during the progress of the reaction.

A suitable tank 14 may be provided in which the liquid titanium tetrachloride may be stored, and from which it may be fed into the reaction chamber 10, as required. A pipeline 16 is provided for this purpose leading from the tank 14 to the cover 12. A suitable valve 18 may also be provided in the pipeline 16 to control the flow of the titanium tetrachloride. The tank 14 may be disposed above the cover 12 and the top of the reaction chamber 10 so that the feeding of the titanium tetrachloride can be accomplished by a simple gravity flow. The pipeline 16, of course, extends throughout the cover 12 into association with the reaction chamber 10.

A suitable melting unit 20 for the magnesium may be provided adjacent the reaction chamber, as shown. This melting unit may be made up of the container 22 for the molten magnesium and the heating jacket 24 which can be electrically heated, for example. The jacket 24 may have a conduit 26 formed therein which extends therethrough from the bottom 28 of the melting unit 20 to the upper surface thereof, where it may continue as the pipeline 30. A heating jacket 32 may be disposed around the pipeline 30, the latter extending through the cover 12 into association with the reaction chamber 10.

The melting unit 20 may also have a pipeline 34 in communication with the cover 36 thereof through which a suitable inert gas, such as argon or helium, may be introduced into the melting chamber above the molten magnesium as desired. Because of this arrangement, the flow of molten magnesium through the pipeline 30 may be controlled by adjusting the gaseous pressure above the surface of the molten magnesium in the melting unit 20. For example, after the magnesium has reached the proper temperature in the melting chamber, the inert gas may be pumped into the chamber to raise the gas pressure therein and to cause the magnesium to flow out through the bottom of the melting unit through the pipelines 26 and 30 into the reaction chamber. It is obvious that by appropriate adjustment of the pressure of the helium within the melting unit above the molten magnesium, the flow of magnesium can be controlled as desired.
As described and claimed in the copending application, Serial No. 349,042, filed April 15, 1953, referred to above, the end of the conduit 30, which is disposed within the conduit member 40, preferably has a vertically disposed portion 38 provided therein, as best shown in Figure 2. At the upper end of this vertically disposed portion, a collar member 40 may be provided within the conduit, which collar member has a centrally disposed opening therein of a diameter considerably smaller than the inner diameter of the conduit 30. The opening 42 within the collar 40 may preferably be substantially cone-shaped, tapering to a relatively small opening in the side of the collar that is adjacent the outlet of the conduit 30 leading into the reaction chamber. This arrangement insures that a relatively fine stream of molten magnesium will be formed at the collar 40, which stream will fall freely therefrom without coming into contact with the inner surface of the conduit 30. This free fall will continue into the reaction chamber until the magnesium reaches the bottom of that vessel or comes into contact with the molten sponge being formed therein.

In carrying out the process disclosed in the copending application Serial No. 353,322, filed May 6, 1953, referred to above, it has been customary to utilize a pipeline or conduit connecting the melting unit and the reaction chamber which has a suitable union therein permitting the reaction chamber and the portion of the pipeline associated therewith to be disconnected from the remainder of the pipeline which is associated with the melting unit. When utilizing such an arrangement, after the desired amount of sponge has been formed in the reaction vessel, the gas pressure within the melting unit is reduced to the value of the pressure in the reaction vessel, or slightly below that point. This will cause the flow of molten magnesium to be interrupted and the magnesium remaining in the pipeline will normally run down in two directions from the highest point therein, thus leaving the pipeline free of magnesium. The temperatures of the various portions of the apparatus are then allowed to fall to room temperature before anything further is done. This cooling takes a relatively long time since the reaction vessel remains inside the electric furnace. The union in the pipeline is subsequently disconnected when the cooling is complete and the reaction vessel, together with its cover and the section of the pipeline attached thereto, is removed from the electric furnace. The titanium sponge present within the reaction vessel is then subjected to further processing.

This is a relatively slow procedure and, in actual production, it would be quite desirable to shorten the period of time required to cool the reaction vessel and its contents. Applicants have devised a means of accomplishing this by providing a liquid trap at the highest point in the pipeline 36 so that a portion of the molten magnesium remains at this point in the pipeline to form a seal, when the magnesium solidifies. As shown in Figures 2, 3 and 4, such a trap may be formed of a plurality of baffle members 44, 46 and 48. These baffle members preferably may be semi-circular in form, as best shown in Figure 4. Two of them, baffles 44 and 48, may be secured by welding, for example, to the bottom of the pipeline in spaced relation while the third baffle member 46 may be attached to the roof of the pipeline substantially midway between the two baffles 44 and 48. As can best be seen in Figure 3, the baffles are of such dimensions that the tip of baffle 46 projects below the upper edges of the baffles 44 and 48. This means that the bottom extremity of the baffle 46 will normally be immersed in the pool 50 of molten magnesium which is adapted to be trapped between the baffle members 44 and 48, as best shown in Figure 2, when the pressure within the melting unit 20 is reduced. The baffle members can be so spaced that they do not present any substantial obstacle to the flow of the molten magnesium when the pressure within the melting unit 20 is sufficient to cause such a flow.

After the pressure has been reduced and the pool 50 has been formed, the portion of the pipeline containing the baffles is cooled and the pool 50 caused to solidify. The union 52 in the pipeline 30 can then be disconnected. This permits the reaction vessel to be removed from the electric furnace within which it is normally disposed during the course of the reaction. Without this improved apparatus, it is substantially impossible to disconnect the union and to simultaneously plug the magnesium pipeline attached to the cover of the reaction vessel rapidly enough to avoid a back diffusion of air from the atmosphere into the reaction vessel. This, of course, is quite undesirable since the reaction vessel and the titanium sponge contained therein are still at a relatively high temperature and any such diffused air will react with the sponge and reduce its quality. The form of lock just described which is provided by the baffle members, however, permits the immediate disconnection of the hot reaction vessel and its removal from the electric furnace without any danger of such contamination of the sponge. The reaction vessel can therefore be removed immediately from the electric furnace and cooled in the open air, which reduces very substantially the amount of time necessary for the cooling period.

It should be understood that the form of lock just described is undoubtedly not the only manner in which this invention can be carried out. It might be desirable, for example, to form a liquid trap in the pipeline 30 by providing a rather abrupt downwardly and upwardly curved portion of sufficient depth so that a plug of magnesium would normally be caused to remain in the lowermost curve thereof, providing a seal substantially in the same manner as previously described in connection with the baffle form of trap.

Likewise it should be mentioned that when other alkali or alkaline earth metals are utilized, such as sodium, potassium and calcium, there is some change in technique necessary. This is brought about by the fact that these metals readily react with the atmospheric gases and moisture. For this reason, it is not desirable to have them exposed to the atmosphere for any substantial length of time. However, any of these metals can be utilized to act as a lock for the relatively short time that it takes to disconnect the union 52 and to attach a suitable cap in its place to the section of the pipeline associated with the reaction vessel which will protect the baffles from such a reactive metal from damage from the atmosphere or moisture.

In the drawing and specification, there has been set forth a preferred embodiment of the invention, and although specific terms are employed, they are used in a generic and descriptive sense only and not for purposes of limitation. Changes in form and in the proportion of parts, as well as the substitution of equivalents are contemplated, as circumstances may suggest or render expedient, without departing from the spirit or scope of this invention as further defined in the following claims.

It is claimed:
1. In an apparatus for preparing a dense sponge of refractory material selected from the class consisting of titanium and zirconium having reaction chamber and a heated container for melting a supply of metal, the combination therewith of a conduit having an inverted U-shaped section comprising a first vertical member connected to said reaction chamber, a second vertical member connected to said heated container, and a generally horizontal member connecting said first and second vertical members, a plurality of stationary baffles disposed in said horizontal member at the highest point thereof and intermediate the ends of said conduit, said baffles including an upper baffle member;
5 extending between and below the outer ends of two upright baffle members so as to entrap a portion of said molten metal to form a seal in said conduit at a point between said reaction chamber and said heated con-
tainer, and a separable joint disposed in said conduit between said baffles and said heated container, where-
by after said horizontal member containing said baffles is cooled, the pool of molten metal solidifies forming an air-tight seal with said baffles, thus permitting said joint to be disconnected, and said reaction chamber to be removed without diffusion of air thereinto.

2. An apparatus for preparing a dense sponge of re-
fractory material selected from the class consisting of titanium and zirconium which comprises an air-tight reaction chamber, a heated container for melting a supply of metal, an arched conduit section between said reaction chamber and said heated container through which the molten metal is adapted to flow, a plurality of baffles disposed in said arched conduit section at substantially the highest point thereof and intermediate the ends of said conduit, said baffles including an upper baffle member extending between and below the outer ends of two upright baffle members so as to entrap a portion of said molten metal to form an air-tight seal in said conduit at a point between said reaction chamber and said heated container, and a separable joint disposed in said conduit between said baffles and said heated container, whereby after said horizontal member containing said baffles is cooled, the pool of molten metal solidifies forming an air-tight seal with said baffles, thus permitting said joint to be disconnected, and said reaction chamber to be removed without diffusion of air thereinto.

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