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METHOD FOR PREPARING VALVE PARTS FOR APPARATUS FOR HANDLING MOLTEN CALCIUM

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This invention relates to valves for an apparatus for handling molten calcium metal and, more particularly, to valves for apparatus for alternately handling molten sodium and calcium metals.

In a copending application of Bernard A. Gruber, Emanuel Gordon and Robert A. Jermain, Serial No. 323,412, filed December 1, 1952, an apparatus is described which is adapted for the recovery of sodium and calcium metals by fractional distillation from the sludge obtained as a by-product in the electrolytic production of sodium metal. This sludge contains about 70 per cent sodium metal and about 20 per cent calcium metal, the balance being essentially chlorides and oxides of these metals present as impurities.

The apparatus described in the above mentioned copending application comprises a vertical distillation retort communicating near its upper end with a downwardly inclined condensation receptacle. The condensing receptacle is provided with a discharge outlet at its lower end which may be opened and closed by a valve having a stationary valve seat and a movable engageable member adapted to be moved to open and close the valve. This valve preferably is of the type having a stationary valve seat and a plug valve adapted to be moved into and out of engagement with the valve seat. The discharge outlet communicates through a conduit with means for separately receiving molten sodium and calcium metals.

The distillation and condensing receptacles as well as the associated parts which are contacted by the hot molten metal or metal vapors are formed of any suitable iron or non-ferrous alloy having good corrosion resistance at high temperatures, such as an iron-chromium alloy, chromium-nickel alloys or chromium-cobalt alloys, stainless steel, etc. It is presently preferred to form the above mentioned movable valve member, such as the plug valve, of type 304 stainless steel containing about 8 to 11 per cent nickel and 18 to 20 per cent chromium. It is convenient to form the valve seat of the same material as that of the body of the condensing receptacle which may be the same or a different material from that forming the movable valve member.

The distillation receptacle is heated to cause the sodium metal to distill from the sludge therein in the presence of an inert gas at a suitable pressure, such as atmospheric pressure. The sodium vapors are condensed to liquid in the condensation receptacle and are caused to pass through the discharge outlet to a sodium metal receiving receptacle.

The valve at the bottom of the condensation receptacle then is closed and the distillation receptacle is heated to cause calcium metal to distill from the calcium-rich residue therein. The calcium vapors are condensed to a solid muff in the condensation receptacle. When the calcium metal has been removed from the calcium-rich residue, the solid muff of calcium metal is melted and the molten calcium collects in the bottom of the condensation receptacle. The valve controlling the discharge outlet of the condensation receptacle then is opened to permit

the molten calcium to flow into a separate calcium receiving receptacle.

When the apparatus above described was first operated, difficulty was experienced with the operation of the valve controlling the discharge outlet of the condensation receptacle. Thus, while the solid muff of calcium metal was being melted, the molten calcium collected in the bottom of the condenser and remained in contact with the valve closing the discharge outlet for a short period of time. When it was attempted to open the valve to permit the molten calcium to flow into its receiving receptacle, it was found that the plug could not be lifted from its seat.

Extensive subsequent investigations were conducted in an attempt to overcome this difficulty. As a result of these investigations, it has been discovered that this difficulty can be overcome by pretreating either the valve seat or the valve plug or both by subjecting the valve part to the action of molten calcium at a temperature below that at which the alloy is substantially corroded but substantially above the temperature at which the molten calcium contacts the valve part in actual use. The period of such pretreatment should be sufficient to cause substantial diffusion of calcium metal into the surface thereof which is to engage the other valve part in use. Thus, the present invention contemplates a method for preparing valve means for an apparatus for handling molten calcium metal at a predetermined temperature by pretreating one or more of the cooperating valve parts with molten calcium at a temperature below that at which the alloy is substantially corroded but substantially above such predetermined temperature for a sufficient period of time to cause substantial diffusion of calcium metal into the surface thereof which is to engage the cooperating valve part.

In the normal operation of the above described distillation apparatus, the pool of molten calcium collecting in the bottom of the condensation receptacle is maintained at a temperature between about 900° to 950°. Consequently, in the practice of the present invention the temperature of the molten calcium used for pretreating one or more of the parts of the valve controlling the outlet of the condensation receptacle should be greater than 950° C. When the valve means includes a plug valve movable toward and away from the valve seat and is formed of type 304 stainless steel containing about 8 to 11 per cent nickel and 18 to 20 per cent chromium, the valve means operates satisfactorily if the plug valve has been pretreated with molten calcium at a temperature of about 1000° C. for a few hours and, preferably for about 24 hours. Pretreatment with molten calcium at a temperature of about 1050° C. tends to corrode this material.

The pretreatment may be conducted in a closed receptacle with an atmosphere of an inert gas under a pressure slightly greater than atmospheric pressure above the molten calcium. After the valve part has been immersed in the molten calcium for the desired period of time, it may be raised and permitted to cool in the inert gas. It then is removed from the receptacle and cleaned as by washing with dilute nitric acid.

I claim:

1. A method for preparing valve means for an apparatus for handling molten calcium at a predetermined temperature, said valve means including a valve seat and a valve plug moveable into and out of engagement with the seat, said seat and plug being formed of an alloy having good corrosion resistance at high temperature, said method comprising pretreating at least one of the valve parts consisting of said seat and plug by subjecting it to the action of molten calcium metal at a temperature below that at which the alloy is substantially corroded but greater than said predetermined temperature for a sufficient period of

time to cause substantial diffusion of calcium metal into the surface thereof which is to engage the other valve part.

2. A method for preparing valve means for an apparatus for handling molten calcium at a predetermined temperature, said valve means including a valve seat and a valve plug moveable into and out of engagement with the seat, said seat and plug being formed of an alloy having good corrosion resistance at high temperature, said method comprising pretreating said plug by subjecting it to the action of molten calcium at a temperature below that at which the alloy is substantially corroded but greater than said predetermined temperature for a sufficient period of time to cause substantial diffusion of calcium metal into the surface thereof which is to engage the valve seat.

3. A method for preparing valve means for an apparatus for handling molten calcium at a predetermined temperature, said valve means including a valve seat and a valve plug moveable into and out of engagement with the seat, said seat and plug being formed of an alloy having good corrosion resistance at high temperature, said method comprising pretreating said seat and plug by subjecting them to the action of molten calcium at a temperature below that at which the alloy is substantially corroded but greater than said predetermined temperature for a sufficient period of time to cause substantial diffusion of calcium metal into the surfaces which are to engage one another.

4. A method for preparing valve means for an apparatus for handling molten calcium at a predetermined temperature, said valve means including a valve seat and a valve plug moveable into and out of engagement with the seat, said seat and plug being formed of an iron alloy having good corrosion resistance at high temperatures, said method comprising forming at least one of the valve parts selected from the group consisting of said seat and said plug of an alloy comprising essentially iron, chromium and nickel, and pretreating the selected part by subjecting it to the action of molten calcium metal at a temperature below that at which the alloy is substantially corroded but greater than said predetermined temperature for a period of time sufficient to cause substantial diffusion of calcium metal into the surface thereof which is to engage the other valve part.

5. The method as described by claim 4 in which the selected valve part is formed of stainless steel having a nickel content of about 8 to 11 per cent and a chromium content of about 18 to 20 per cent by weight.

6. A method for preparing valve means for an apparatus for handling molten calcium at a predetermined temperature, said valve means including a valve seat and a valve plug moveable into and out of engagement with the seat, said seat and plug being formed of an iron alloy having good corrosion resistance at high temperatures, said method comprising forming said plug of an alloy comprising essentially iron, chromium and nickel, and pretreating said plug by subjecting it to the action of molten calcium metal at a temperature below that at which the alloy is substantially corroded but greater than said predetermined temperature for a period of time sufficient to cause substantial diffusion of calcium metal into the surface thereof which is to engage the other valve part.

7. The method as described by claim 6 in which the said plug is formed of stainless steel having a nickel content of about 8 to 11 per cent and a chromium content of about 18 to 20 per cent by weight.

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