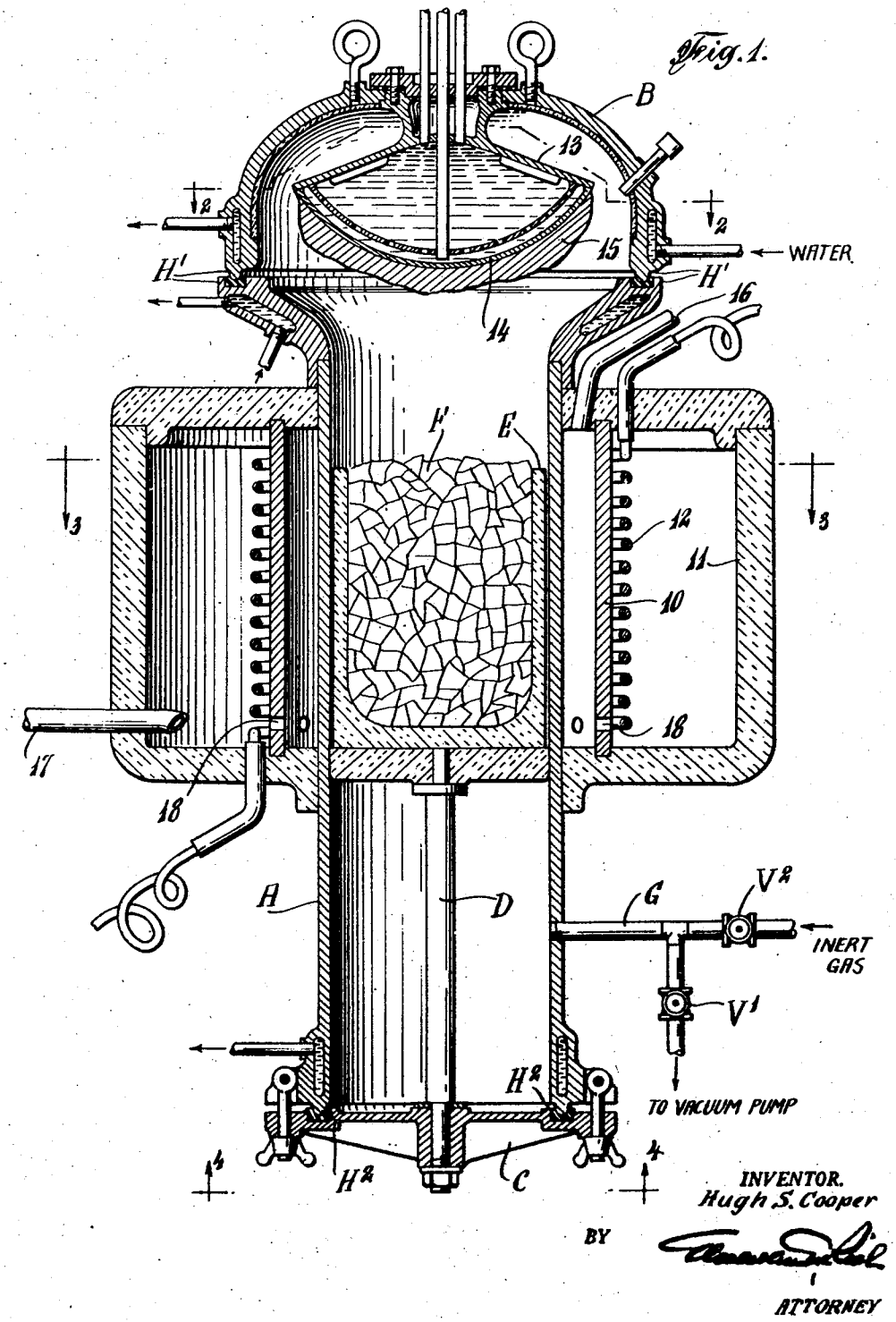
Oct. 28, 1947. H. S. COOPER 2,429,668

METALLURGICAL FURNACE AND CONDENSER

Filed Aug. 7, 1942 3 Sheets-Sheet 1



INVENTOR.
Hugh S. Cooper

BY

ATTORNEY

Oct. 28, 1947. H. S. COOPER 2,429,668

METALLURGICAL FURNACE AND CONDENSER

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Fig. 2.

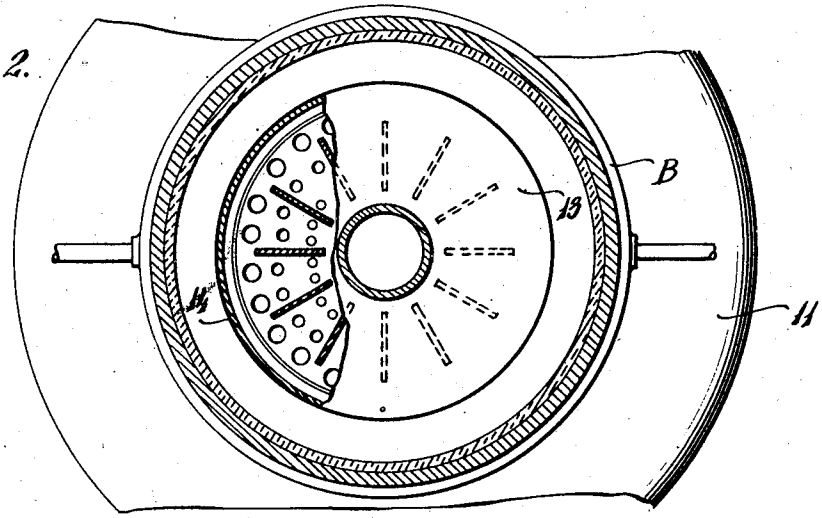


Fig. 3.

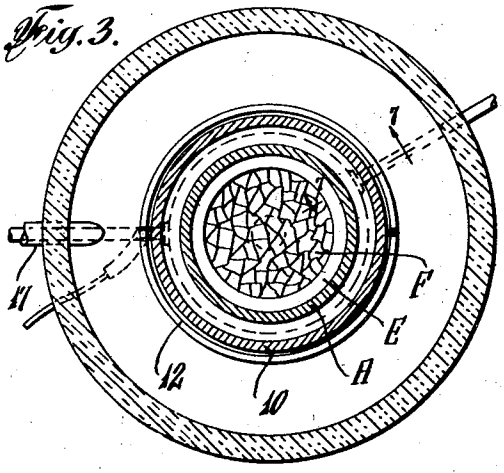


Fig. 4.

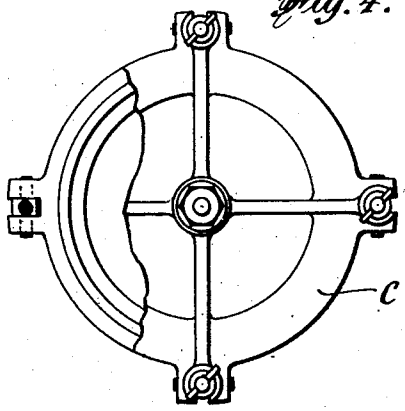


Fig. 7.

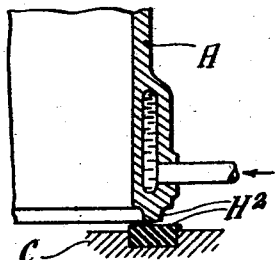
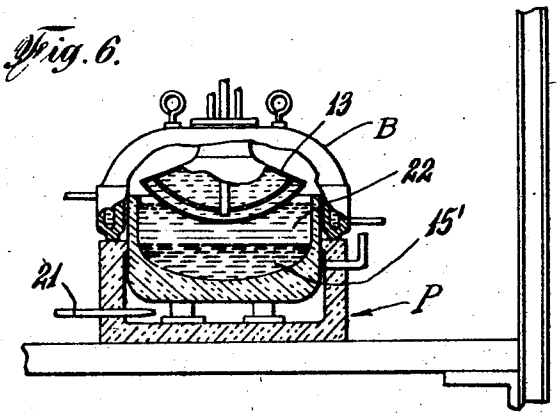


Fig. 6.



INVENTOR.
Hugh S. Cooper
BY
ATTORNEY

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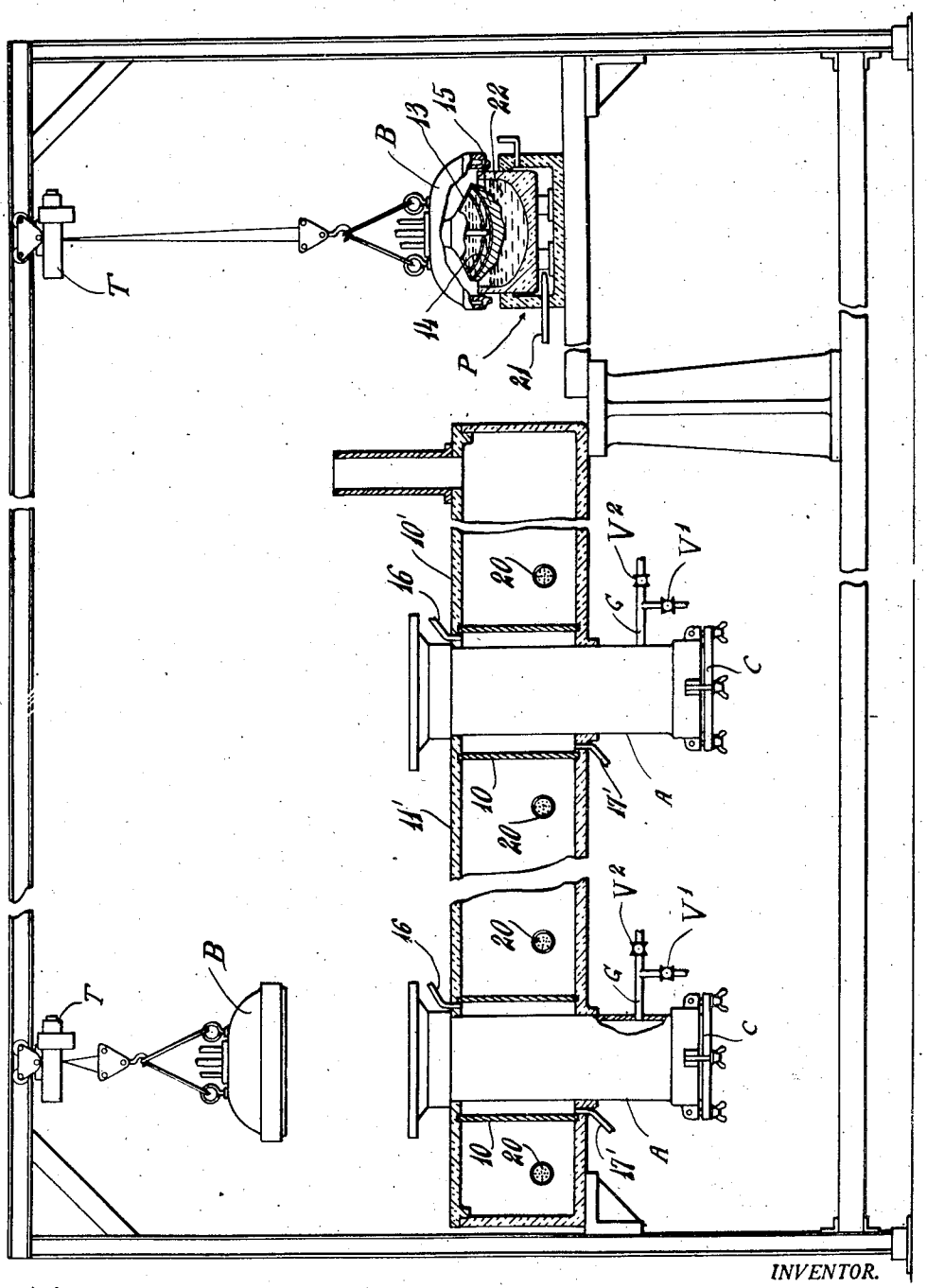


Fig. 5.

INVENTOR.
Hugh S. Cooper
BY
ATTORNEY

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UNITED STATES PATENT OFFICE

2,429,668

METALLURGICAL FURNACE AND CONDENSER

Hugh S. Cooper, Cleveland, Ohio, assignor, by direct and mesne assignments, to Acme Aluminum Alloys, Inc., Dayton, Ohio, a corporation of Ohio

Application August 7, 1942, Serial No. 453,984

9 Claims. (Cl. 266—17)

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This invention relates to metallurgical furnaces and more particularly to apparatus adapted for the reduction of metal oxides at reduced pressures and for the vacuum distillation and condensation of the metal constituent of said oxides.

One of the objects of the present invention is to provide an improved apparatus for the reduction of metal oxides under reduced pressures.

Another object is to provide an improved apparatus for the reduction of metal oxides under reduced pressures and for the distillation and condensation of the metal constitutent of the said oxides.

Another object is to provide an improved apparatus in which to practice the reduction of magnesium oxide by a high boiling point reducing agent at reduced pressures at temperatures above the vaporization temperature of magnesium, with resultant vaporization and condensation of the magnesium at a point removed from the temperature zone of reduction.

Still another object is to provide an improved apparatus for the production of metallic magnesium.

Other objects will be apparent as the invention is more fully hereinafter disclosed.

In accordance with these objects, I have devised the metallurgical apparatus illustrated in the accompanying drawings, of which the following is a full and complete description:

In the drawings:

Fig. 1 is a sectional side elevational view of the apparatus of the present invention;

Fig. 2 is a sectional view taken along plane 2—2 of Fig. 1;

Fig. 3 is a sectional view taken along plane 3—3 of Fig. 1;

Fig. 4 is a bottom view along plane 4—4 of Fig. 1;

Fig. 5 is a side elevational view partly in section illustrating one practical mode of applying the apparatus of Fig. 1 commercially;

Fig. 6 illustrates one feature of the present invention, and

Fig. 7 illustrates a second feature of the present invention.

In the production of magnesium by a process involving the reduction of its oxide by a metallic reducing agent at reduced pressures and at temperatures above the vaporization temperature of the magnesium, but below the vaporization temperature of the metallic reducing agent, the operations of reduction, volatilization of magnesium

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and condensation of magnesium must be performed in a closed and evacuated container.

One of the essential difficulties in this general method of reducing magnesium and other metal oxides is to find and employ a metallic reducing agent which will effect the reduction of the magnesium oxide at a commercially practical temperature within the range of temperatures easily obtained and maintained by known heat generating equipment without the use of special heat-resistant materials. In co-pending application Serial No. 453,051 filed July 31, 1942, now Patent 2,387,979 issued Oct. 30, 1945. I have disclosed such a metallic reducing agent, namely, calcium silicide ($CaSi_2$) which is effective as a reducing agent for magnesium oxide at temperatures within the range 900–1150° C. at reduced pressures below about 1 millimeter of mercury.

Another essential difficulty is to effect the condensation of the magnesium vapors in such a manner as to promote the formation of large grains or masses of sufficient size as to resist spontaneous combustion when the exposed surfaces thereof are subjected to oxidation. In co-pending application Serial No. 435,678 filed March 21, 1942, now Patent 2,351,489, I have disclosed apparatus designed for use primarily in the vacuum reduction and distillation of magnesium which is featured by the use of a depending water-cooled condenser disposed interiorly the apparatus in relatively close spaced relation to the point of origin of the magnesium vapors which provides for the condensation of the magnesium vapors on a water-cooled surface thereof at a temperature approximating but below the freezing or solidification temperature thereby obtaining grain growth of the deposited magnesium vapors into crystalline structures having a large mass relative to its surface area, which protects the deposited magnesium from being heated up on surface oxidation to a temperature at which it will vaporize and spontaneously combust.

The present apparatus combines this feature of my said co-pending application Serial No. 435,678 with other features, in the design of a vacuum reduction and distillation and condensing furnace, that is adapted for wide utility in the art in the reduction of substantially all metal oxides whose metallic constituent may be vaporized at low pressures at temperatures up to about 1500° C., but which in the specific embodiment shown, is specifically designed for the reduction of magnesium oxide by calcium silicide at temperatures within the range 900–1150° C.

Referring to the drawings, the vacuum reduc-

tion-distilling-and condensation apparatus of the present invention comprises an elongated vertically sustained chamber A, preferably circular in cross-section, having a portion of its length intermediate the ends thereof enclosed by a means M to heat the same to any desired temperature. In the specific embodiment shown in Fig. 1, means M comprises a tubular refractory member 10 sustained within refractory enclosure 11 in spaced relation to the outer surface of tubular chamber B and electrical resistance heating element 12 disposed adjacent the surface of tubular refractory member 10 opposite to the face lying in spaced relation to the outer surface of tubular chamber A. Any alternative means for heating chamber A in substantially the same manner may be substituted for the means shown, without departure from the present invention, but the means shown is preferred as will be more fully hereinafter disclosed.

The upper end of chamber A is provided with a removable condensing chamber B and the bottom end of the chamber A is provided with a detachable closure means C. Means D which is essentially a platform also is provided to sustain a refractory container or crucible E filled with a charge consisting of a briquetted mixture of MgO and $CaSi_2$ in approximately equivalent molecular weight proportions, in heating position within the heated portion of chamber A and means, such as conduit G communicating to the interior of chamber A, is provided through valve V_1, to lower the pressure within chamber A or through valve V_2, to introduce any desired gas within the chamber A.

It is preferable to provide means, such as water-cooled vacuum seals H_1—H_2 to removably and to detachably secure condensing chamber B and closure means C, respectively, in position on the opposite ends of chamber A.

The specific size, shape and configuration of condensing chamber B and closure means C may be varied widely without essential departure from the present invention, as one skilled in the art may readily perceive; as may also the specific arrangement provided by means D to sustain crucible E within the heated portion of chamber A.

With the arrangement shown in Fig. 1, however, the means provided is adapted to accomplish the desired result, namely, the heating of charge F contained within crucible E to a temperature, at the low pressure provided, at which the reduction reaction, resulting in the formation of a vaporizable metal (Mg), proceeds with simultaneous vaporization of the metal and condensation of the metal vapors upon the surface 14 of water-cooled condenser 13 which is dependingly sustained interiorly within condenser chamber B with its condensing surface 14 disposed in spaced and covering position over crucible E, so as to provide a relatively short path of travel of the metal vapors from the crucible to the condensing metal 15 collecting thereon at a temperature at which the deposited metal vapors undergo grain growth into relatively large sized masses, by the heat energy radiated thereto from the heated crucible.

The major advantage of the arrangement shown is that a plurality of crucibles E may be heated successively within chamber A without removing condenser chamber B from closure position on the upper end of chamber A. This is obtained by closing valve V_1 and opening valve V_2 filling the chamber A with a relatively light inert gas, such as non-explosive mixture of hydrogen and nitrogen or helium, to a pressure approximating atmospheric pressure which releases vacuum seal H_2 on closure member C permitting the removal of the member and the removal of crucible E from chamber A and the insertion of a second crucible E filled with fresh charge F in heating position within the chamber A and a reseating of closure member C in closure position on the end of chamber A without subjecting the condensed metal 15 to oxidation and without interrupting the heating of the heated portion of chamber A.

While the second crucible and its contents are heating up to the reaction temperature the gas pressure contained therein may be reduced to the desired pressure by closing valve V_2 and reopening valve V_1.

The release of vacuum during the re-charging operation fails to break the hermetic seal normally provided by the weight of condensing chamber B bearing on vacuum seal H_1 and, accordingly, the thickness of condensed metal 15 on condenser surface 14 may be built up by successive reduction and distilling operations to the extent desired.

The essential structural features of condenser chamber B and of vacuum seals H_1 and H_2 are described and claimed in my copending application Serial No. 435,678, above identified, and, per se, form no part of the present invention, being adapted to perform substantially the same functional result in the present apparatus as described and claimed therefor in my said co-pending application. It is believed unnecessary to specifically describe the same in view of the clear illustration thereof in the drawings. However, attention is directed to the fact that vacuum seals H_1 and H_2 each consist of annular resilient members and annular extensions disposed in engaging position oppositely to each other on the opposite parts which it is desired to vacuum seal together. The mechanical engagement of the two annular members, either by the force of gravity as in the case of the joint between the condensing chamber B and chamber A or mechanically as indicated in the case of closure member C on the bottom end of chamber A, operates to form an hermetic seal therebetween, which seal becomes a vacuum tight seal with decrease in pressure in the interior of chamber A as a result of the movement of the two closures B and C against the upper and lower ends of chamber A, incident to the gas and air pressure differential between the inside and outside of chamber A.

One marked advantage of the arrangement shown for heating chamber A lies in the feature of providing tubular refractory member 10 adjacent the outer surface of the chamber A, but in spaced relation thereto. The problem solved by this arrangement is primarily economic.

Heretofore in the art of furnace construction, such as in muffle, tube furnaces, and the like, where it is desired to heat a metal tube or container to elevated temperatures, it has been necessary to employ high temperature oxidation resistant alloys for the metal tube or container. These alloys are expensive, even in normal times, and at the present time are difficult to obtain, due to the scarcity of the essential alloying constituents, such as chromium, nickel, manganese and the like.

However, there are many other steels which, while lacking in high temperature oxidation resistance, nevertheless when protected from oxidation possess the requisite high temperature

strength to withstand continued or repeated heating to relatively high temperatures under the stress conditions imposed by the pressure differentials existing in the present apparatus.

In the heating arrangement shown, the exterior surface of the heated portion of chamber A is protected from oxidation, by means of a protective reducing gas, such as H_2, cracked ammonia gas, and the like, by circulating such gases within the space gap between tubular refractory member 10 and the surface of chamber A. As most of these gases will burn or oxidize when discharged in the heated state into air, means such as outlet discharge conduit 16 is provided to conduct the gases flowing through the space gap to a point remote from the apparatus for burning or for reclaiming, as may be desired.

Where the means for heating tubular refractory member 10 and the enclosed length of chamber A to the desired temperature comprises the electrical resistance heating coil or element 12, it is preferable to utilize this protective gas to protect the incandescent metal of the heating coil from oxidation also. In the arrangement shown in Fig. 1, the protective gas is introduced within the refractory enclosure 11, which is sealed from the atmosphere in any convenient manner well known in the art, by means of pipe 17, filling the enclosure and surrounding heating element 12 protecting the same from oxidation and passing through openings 18 in the bottom of tubular refractory member 10 into the space gap between this member 10 and the wall of chamber A enclosed thereby, and thence out through conduit 16 to the discharge point. By tightly sealing the joint between chamber A and the top and bottom walls of refractory enclosure 11, the entire enclosed length of chamber A thus is protected from oxidation.

When electrical resistance heating is dispensed with and means, such as gas or oil fired burners are employed, as is also common in the art, it is preferable to omit holes 18 in the bottom of refractory member 10 and to introduce the protective gas directly into the space gap through tubes 17', as indicated in Fig. 5.

Another advantage of the present invention, resides in the production possibilities obtained through the use of a battery of such reduction, distillation and condensing furnaces, illustrated in Fig. 1 in combination with a melting furnace. Referring to Fig. 5, these advantages will be apparent.

In the arrangement shown, a plurality of furnaces, such as shown in Fig. 1 may be sustained in spaced relation in a refractory enclosure 11' which is heated interiorly by a plurality of gas or oil fired burners 20, and a transfer means T may be provided to remove any one of the condenser chambers B therefrom and to convey the same to a position where it may be lowered into closure position over melting down furnace P with the condensed metal 15 on condenser surface 14 immersed in a molten bath 22 of flux maintained, as by burner 21, at a temperature above the melting point of the condensed metal 15, or 15' as shown in Fig. 6.

This arrangement is particularly adapted in the production of magnesium metal in accordance with the invention of my co-pending application Serial No. 453,051.

Before condenser chamber B is removed from closure position over chamber A, it is, of course, necessary to release the vacuum by filling the chamber A with inert gas as hereinabove described in connection with the recharging operation. The introduction of an inert gas in chamber A normally does not break the hermetic seal connection between the engaging annular parts of seal H_1, however, as the pressure within container A becomes equal to atmospheric pressure container B may be readily removed from its closure position on the end of chamber A, as by the vertical displacement of condenser chamber B upwardly by the means T. Any convenient means may be employed for this purpose, the means, per se, forming no part of the present invention.

Where continuous operation of the apparatus is desired, a second chamber B may be immediately disposed in closure position on the end of chamber A, and chamber A may be recharged as hereinabove described following a short heated cycle in the presence of an inert gas to remove water vapor and air from chamber B.

It is believed apparent from the above description and accompanying drawings that the present invention may be widely modified without essential departure therefrom and all such modifications and adaptations are contemplated as may fall within the scope of the following claims.

What I claim is:

1. A metallurgical furnace for the vacuum production of readily vaporized metals such as magnesium, said furnace comprising an elongated vertically sustained metallic container open at both ends, means to heat an extended length of the container intermediate the ends to elevated temperatures, water cooling means to maintain the open ends of said container at relatively low temperatures, top and bottom closure members for the open ends of said container hermetically seated and sustained, respectively, in closure position on the said open ends, the top said closure member being provided with an interiorly depending water cooled condenser chamber, means to sustain a crucible within the heated portion of said container, and means to obtain and maintain a desired low pressure of an atmosphere within the interior of the container.

2. The furnace of claim 1, wherein means is provided to prevent oxidation of the heated exterior surface of said container, said means comprising a tubular refractory body disposed in spaced relation to the said heated surface and means to flow a reducing and nonoxidizing gas under pressure through the space gap between said refractory body and the exterior heated surface of the said container.

3. In combination, a plurality of furnaces of the type described and claimed in claim 1, sustained vertically in spaced relation and at least one melting furnace, said melting furnace containing a bath of molten flux heated to a temperature approximating the melting point of the vaporizable metal produced in the said metallurgical furnaces and condensed upon the interiorly depending water cooled condenser of the top closure means thereof, the open end of said melting furnace being provided with means to sustain the said top closure means of the said metallurgical furnaces in closure position thereon with the said interiorly depending condenser means thereof substantially immersed in the said molten flux, and transfer means to move the said top closure members from closure position on the said metallurgical furnaces to closure position on the said melting furnace and vice versa.

4. In a metallurgical furnace, the combination of an elongated vertically sustained tubular metal

container, means to heat a portion of its length intermediate its ends to elevated temperatures, means to maintain the open ends of the container at relatively low temperatures, and means to protect the heated exterior surface of the container from surface-oxidation, said means comprising a tubular refractory body sustained in determined spaced relation to the heated surface of the container and means to flow a reducing and non-oxidizing gas under pressure through the space gap therebetween.

5. In a metallurgical furnace for the production of vaporizable metals under reduced pressure, an elongated vertically sustained tubular metallic container open at both ends and provided at each said end with water cooling means to maintain the temperatures of said open ends at approximately atmospheric temperatures, the upper said end being provided with an upwardly facing area having an annular recess therein with an annular gasket of resilient material seated therein and the said bottom end being provided with a depending annular extension, a vaulted closure member for the upper said end, said member having an interior depending water cooled condenser and being provided with an annular extension on the bottom thereof to engage and seat upon the said annular gasket, a closure member for the bottom end of said container, said member being provided with an annular recess in the face thereof containing an annular gasket of resilient material to engage and seat upon said annular extension on the said bottom end of the container, means removably securing the said bottom closure member in closure position upon the bottom end of said container, means to heat a portion of the length of said container intermediate the ends thereof to elevated temperatures, means to sustain a crucible within said heated portion of said container, and means to provide a desired reduced pressure of a gas atmosphere within said container.

6. Metallurgical apparatus for the production under reduced pressures of vaporizable metals such as magnesium, said apparatus comprising in combination an elongated vertically sustained tubular metal container, means to heat an extended length of said container intermediate the ends thereof to elevated temperatures above about 900° C., means to protect the exterior surface of the said heated portion from oxidation, said means comprising a tubular refractory body sustained in determined spaced relation to the heated surface of said container and means to flow a reducing and non-oxidizing gas under pressure through the space gap therebetween, means to water-cool the opposite open ends of said container, an annular gasket consisting of resilient material disposed in an annular recess in an upwardly facing surface of the upper end of the container and a vaulted closure member for the said upper open end of the container, said member being provided with an annular rib extension on the bottom face thereof to engage and seat upon said annular gasket in the said upper end of the container and with an interiorly depending water cooled condenser surface, a depending annular rib extension on the bottom open end of the said container, a closure member for the said bottom end, said member being provided with an annular recess and with an annular gasket consisting of resilient material seated in said recess to engage and seat upon said annular rib extension depending from the said bottom end of the container, means to removably secure the said bottom closure member in closure position on the open end of said container, said means providing substantially free vertical movement of the closure member upwardly from its secured position, means to sustain a crucible in position within said heated portion of said container, and means to introduce a desired low pressure of an atmosphere within the container.

7. In a metallurgical furnace for the production of vaporizable metals under reduced pressures, the combination of an elongated vertically sustained metallic container open at both ends and provided at each said open end with water cooling means to maintain the temperatures of said ends at approximately atmospheric temperatures, an annular gasket consisting of resilient material seated in an annular recess in the upper face of the upper water cooled end of said container, an annular rib extension depending from the bottom water cooled end of said container, means to heat an extended length of said container intermediate the said water cooled ends of the container, means to protect the exterior surface of said heated portion from oxidation, said means including a tubular refractory body located in spaced relation to the said exterior surface of said heated portion and means to flow a reducing and non-oxidizing gas under pressure through the space gap therebetween, a vaulted closure member for the upper water cooled end of said container, the open end of said closure being provided with an annular rib extension adapted to engage and to seat upon the annular gasket in the upper face of the said upper water cooled end of the container and being provided with an interiorly depending water cooled condenser chamber, a closure member for the bottom water cooled end of said container, said closure member being provided with an annular gasket consisting of resilient material seated in an annular recess therein in a position adapting the same to engage the annular extension on the water cooled bottom end of said container, the said bottom end of the container and the said bottom closure member being provided with cooperating mechanical means to detachably sustain the said bottom closure member in closure position on the bottom end of the container to be movable vertically upward in response to pressure differentials between the atmospheres interior and exterior the container, means to removably sustain a charge containing crucible within the said container within the heated portion of the length thereof, and means to provide a desired reduced pressure of an atmosphere within said container when the said end closure members are located in closure position on each water cooled end of said container.

8. In apparatus for the production of vaporizable metals, such as magnesium, the combination of a plurality of vertically sustained vacuum furnaces, each provided with a vaulted condenser chamber seated in removable closure position upon the open upper ends of said vacuum furnaces, each said chamber being provided with an interiorly depending water cooled condenser, and at least one melting furnace provided with an open end adapted to receive in closure position the said vaulted condenser chambers with the said interiorly depending water cooled condenser thereof disposed within the melting furnace and substantially immersed in a bath of molten flux in said melting furnace, and transfer means to move the said vaulted condenser chambers from closure position on the said vacuum furnace to

closure position on the said melting furnace and vice versa.

9. Metallurgical apparatus for the production of vaporizable metals, such as magnesium, said apparatus comprising in combination a plurality of metallurgical furnaces each consisting of an elongated tubular metal container each of substantially the same diameter, each said container being sustained vertically and being provided with means to heat a portion of the length thereof intermediate the open ends thereof to elevated temperatures and with top and bottom closure members detachably secured in vacuum tight relation to the upper and lower open ends thereof with the top said closure member being vaulted and provided with an interiorly depending water cooled condenser, means to sustain a crucible interiorly of each said container within the heated portion thereof and means to maintain a desired reduced pressure within said container, and at least one melting furnace, said melting furnace consisting of a container having at the top thereof an outside diameter approximating the inside diameter of the vaulted top closure members of the said metallurgical furnaces and an outer annular support means in a position to sustain the said closure member in closure position over the said melting furnace with the depending condenser thereof substantially immersed in a bath of molten flux contained in the melting furnace, and transfer means to remove the said top closure member from closure position on said metallurgical furnaces to closure position on said melting furnace and vice versa.

HUGH S. COOPER.

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