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MANUFACTURE OF MAGNESIUM

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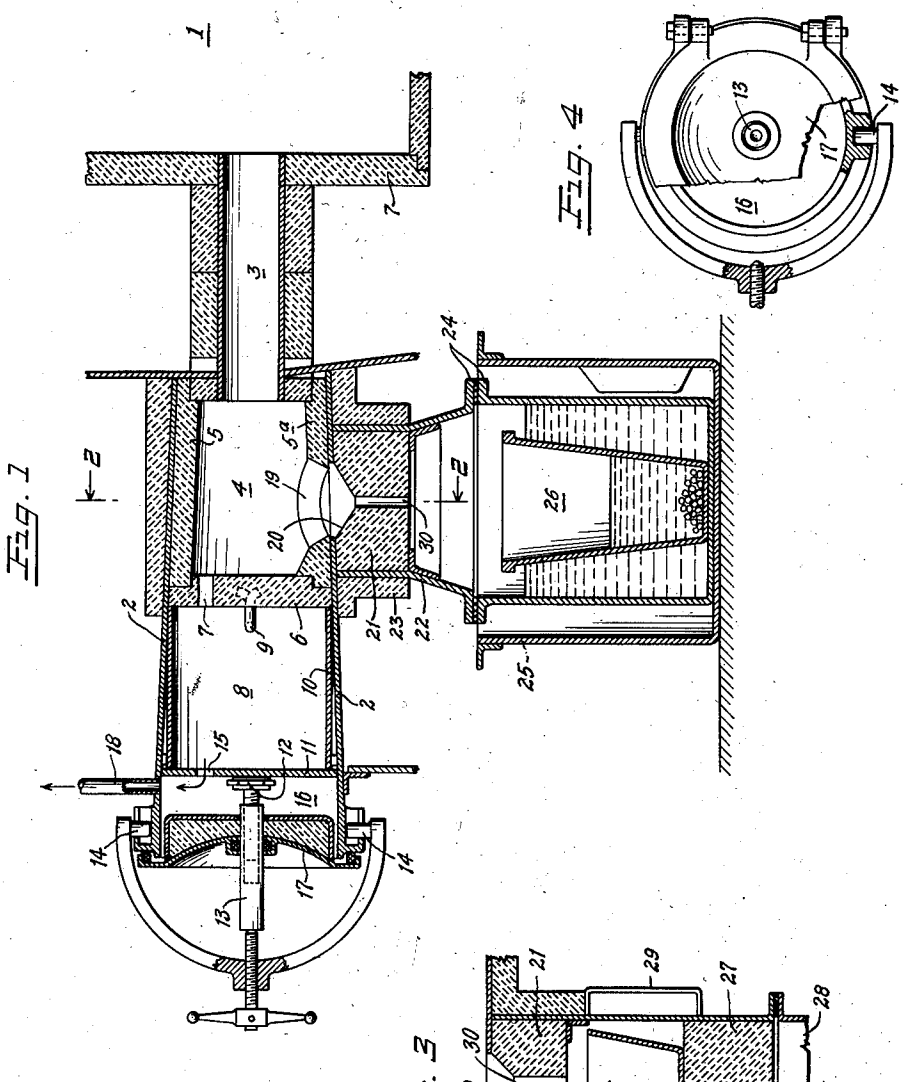


Fig. 1



Fig. 4

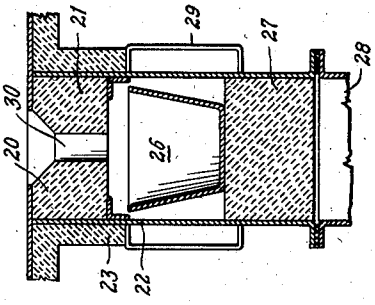


Fig. 3

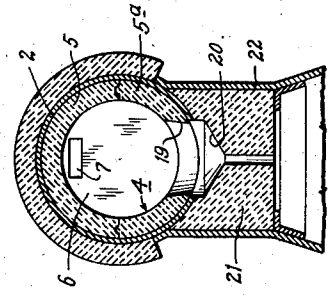


Fig. 2

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## MANUFACTURE OF MAGNESIUM

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13 Claims. (Cl. 266—19)

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My invention relates to the production of magnesium by thermal reduction of an ore of that metal, such as dolomite. Dolomite mixed with a charge of a reducer such as ferro-silicon, is submitted to electrical heating. Such reaction is accomplished within a furnace under vacuum, to avoid the combustion of the magnesium when and as it appears or its transformation into oxides, nitrides and other compounds. The magnesium vapors are condensed in an enclosure connected to the furnace.

To obtain the best yield for the reduction of the ore, it is customary to maintain in the furnace a high degree of vacuum under which the magnesium vapors will condense to the solid state. In that case, the reduction operation must be followed by a fusion of the condensed magnesium in order to obtain a compact ingot or mass.

When it is desired to obtain magnesium in the liquid state directly, that is to say to condense to the liquid state the vapors issuing from the reduction enclosure, the condenser and consequently the reduction furnace must be under a pressure somewhat higher than for the usual condensation to the solid state. But in such a case, there is a risk of an insufficient yield for the reduction operation and also of magnesium vapors being driven along into the vacuum pump piping where they may, when condensing, cause prejudicial clogging.

My invention has for its object to provide an efficient method for obtaining magnesium directly in the liquid state while obviating the above mentioned drawbacks.

The method according to my invention consists in providing in the condenser two successive communicating zones, the first zone communicating with the reduction enclosure and the second zone with the vacuum pump, in establishing in said zones temperature and pressure conditions different from one zone to the other and adapted for the first zone to assure the condensation of magnesium vapors to the liquid state and for the second zone the condensation to the solid state, the communication between the two zones being arranged to create (for instance by means of a throttle) in the gaseous current sucked by the pump a loss of pressure capable of maintaining in the first zone an excess of pressure with respect to the second zone.

Under those conditions, the vacuum pump is in direct communication only with the zone of the condenser where a comparatively low temperature prevails, corresponding to a very low

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vapor tension of the magnesium, such zone consequently condensing and practically retaining all of the magnesium and avoiding the passage of vapors of that metal into the vacuum pump.

As to the reduction of the ore, though it takes place under pressure conditions enabling a condensation to the liquid state, it is nevertheless accomplished with a satisfactory yield as such pressure is very little above the minimum required for obtaining the condensation to the liquid state. Such minimum value is comparatively low; and the pressure in the reduction furnace differs from the high degree vacuum prevailing in the zone of condensation to the solid state only by the small value corresponding to the loss of pressure produced in the vapors current between the first and the second zones in the condenser. It is thus possible to maintain the pressure in the reduction furnace at a value low enough to have a good yield, while such pressure is sufficient to ensure the condensation to the liquid state in the first zone of the condenser.

The method according to my invention may be carried out by maintaining the first zone of the condenser at a temperature of the order of 660° C. and at a pressure of the order of 2 millimeters of mercury, while the second zone is maintained at a temperature slightly lower than 400° C. and at a very low pressure equal to the tension of the magnesium vapors at that temperature.

A particularly advantageous embodiment of my invention consists in providing small dimensions for the condenser in the first zone of condensation to the liquid state, and in continuously eliminating the condensed liquid magnesium when and as it is formed. To that end, the compartment of the condenser where the magnesium vapors condense to the liquid state communicates at its lower part with a fluid tight chamber containing an ingot mould into which the liquid metal falls.

At its upper part the first zone for the liquid condensation communicates with the second zone for the solid condensation through an aperture of small section which is consequently able to cause, on the path of the gases passing from the first to the second zone, a loss of pressure equal to the difference of pressure which it is desirable to establish from one zone to the other.

In the zone for the liquid condensation the pressure and temperature must correspond to the indications of the phase diagram for the

liquid phase, i. e. a temperature and pressure slightly higher than the triple point.

The temperature, controlled by a pyrometer, is maintained at the desired value by any suitable means.

Under those conditions, the vapor streamlets reaching the partition separating the first zone of liquid condensation from the second zone of solid condensation are condensed when and as they arrive and the liquid falls into the ingot mould placed in the fluid-tight chamber which is under the same pressure or vacuum conditions as the first zone with which it communicates.

The metal may settle in the ingot mould which is either at ordinary temperature or hot.

In case it settles in an ingot mould at ordinary temperature, only the discharging pipe supplying the liquid to the ingot mould is maintained at the temperature of the condenser zone of liquid condensation, the chamber where the ingot mould stands being left at the ordinary temperature. The ingot mould contains a certain quantity of oil and is also surrounded by an oil bath so that the liquid drops settle in the oil of the ingot mould, in the form of small balls separated from their impurities.

The oil surrounded the ingot mould is provided for the sake of precaution, in case some streamlets of metal vapors would exceptionally pass through the discharging orifice and penetrate into the chamber of the ingot mould. They will then condense in the oil which is the coldest point of the chamber containing the ingot-mould.

If the metal settles in a hot ingot mould, said ingot mould is laid on a base made of insulating material under which is provided a fluid-tight cooled door. All the upper part and the insulating bottom of the ingot mould chamber are carefully heat insulated or even reheated if necessary, so that the ingot mould will be at the same temperature as the condenser zone of liquid condensation. In that case the liquid flowing into the ingot mould is maintained liquid.

Before taking the ingot mould out of the chamber, the heat-insulating material or the heating means surrounding the ingot mould chamber is taken off and replaced by water-jackets which in a few minutes produce the solidification of the metal in the ingot mould. It is then sufficient to open the door at the lower end of the chamber to take off the ingot mould and the solidified metal which it contains.

The oil is filtered, if necessary, before being used again.

The accompanying drawings show merely as an example an embodiment of a condenser improved according to my invention.

Fig. 1 is a vertical view, with an axial section of the complete condenser.

Fig. 2 is a cross section along line II—II of Fig. 1.

Fig. 3 is an axial section of a modified design of the ingot-mould chamber.

Fig. 4 is a front view, partly broken away, of the door to the condensing chamber, showing the yoke swung aside.

The furnace in which the reduction of the charge takes place (by electrical heating under vacuum) may be of any suitable type and for that reason it is neither shown on the drawings nor described.



The reaction enclosure 1 of that furnace is communicating at one of its ends with the condenser comprising for instance, an iron sheet envelope 2 shaped as a truncated cone, having the same horizontal axis as the duct 3 of refractory material connecting the condenser with the reaction enclosure.

According to the invention, in the part of the condenser adjacent to the furnace there is provided a chamber 4, called liquefaction chamber, constituted by refractory walls 5, 5a and 6, and directly communicating with the reaction enclosure of the furnace through the duct 3. At its end opposite to duct 3, the chamber 4 comprises a refractory wall 6 tightly set on the lateral walls 5 and 5a and through which there is provided a duct 7 of very small section, connecting the chamber 4 with the extremity of the condenser which constitutes the crystallization chamber 8. The wall 6 plays the part of a door giving access to the liquefaction chamber; it is provided with a handle 9 or any other grasping means, and it is maintained in its place by a sheet-iron casing 10 constituting the inner wall of the crystallization chamber. Such casing 10 comprises an end-plate 11, carefully heat-insulated, which at the center bears a fastening clamp 12 placed at the end of a screw 13 that is engaged with the screw threads of a fork-link, the ends 14 of which bear on the wall 2 of the condenser.

The screw 13, fork link, and door 17 are not claimed herein, and there is not represented the reheating or cooling means which may permissibly be used for maintaining the temperature at about 400° C. inside the crystallization chamber 8. Such chamber communicates, through the small hole 15 provided in the end-plate 11, with the back capacity 16 closed by a fluid-tight door 17, and communicates also with the vacuum source through the duct 18.

The liquefaction chamber 4, the cross section of which is circular as shown on Figure 2, comprises at its lower part an aperture 19 above a funnel-shaped cavity 20 provided in a refractory block 21 set in a sheet-iron casing 22 welded with the wall 2 of the condenser, outwardly insulated at 23 along the height of block 21 and permissibly provided with reheating means. The chamber containing the ingot mould is placed under the casing 22 to which it is tightly connected by flanges 24. It is surrounded by a jacket 25 in which cooling water is circulated. The ingot mould 26 is immersed in an oil bath, as above explained.

Such arrangement is suitable for the case where the metal formed in the liquid state in the liquefaction chamber 4 is gathered in solid form in the ingot-mould 26. The jacket 25 enables to maintain in the ingot mould chamber a temperature which is sufficiently low so that the liquid metal flowing through the pipe 30 will solidify when reaching the ingot mould 26. The latter may advantageously contain oil into which the liquid drops fall and solidify in the shape of small balls after having been separated from their impurities.

When it is desired that the formed liquid metal should fall into the ingot mould and be maintained there in the liquid phase during all the operation, it is possible to use the arrangement shown on Figure 3 where the ingot mould is immediately adjacent to the block 21 and is laid for instance on a base constituted by a refractory and insulating mass 27. The ingot mould cham-

ber is closed at its lower part by a bottom 28 with a fluid-tight joint which may, if required, be cooled by any suitable means. The chamber containing the ingot-mould is maintained at the temperature of the liquefaction chamber by insulating means or by reheating means if necessary. To that end insulated semi cylinders may be provided, being permissibly equipped inside with heating resistances. Before the taking out of the mould, at the end of the operation, the cylinders 29 are taken off and they are replaced by two semi circular sheaths where a cooling liquid such as water is circulating. The liquid metal of the ingot mould is solidified and the operation is ended by breaking the vacuum in the furnace and taking off the ingot mould. At the same time, the casing 10 is taken away and it is rapidly introduced into a cooled, tightly closed enclosure of suitable shape, where the vacuum is established so that the gathered crystals may cool without being subjected to the action of air so as to avoid any loss of metal.

Of course the invention which was described principally in respect of its application to the case of magnesium is applicable to the production, in similar conditions, of metals which are obtained by condensation of their vapors.

What is claimed is:

1. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, a throttle being provided in the communication between the two zones to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone.

2. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the said first zone being separated from the second zone by a partition provided at its upper part with an orifice to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone.

3. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors



to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state.

4. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, and means to maintain the upper part of said chamber provided with said orifice through which the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser.

5. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, and means to maintain the upper part of said chamber provided with said orifice through which the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser, the lower part of said chamber being maintained at a suitable temperature to have the metal deposited in the ingot mould in the solid state.

6. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the re-

action chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, and means to maintain the upper part of said chamber provided with said orifice through which the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser, the other part of said chamber being heat insulated during the discharge of the liquid metal to the ingot mould and the filling of said mould with the liquid metal.

7. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, and means to maintain said chamber at the same temperature as the first zone of the condenser during the discharge of the liquid metal to the ingot mould and the filling of said mould with the liquid metal.

8. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, means to maintain the upper part of said chamber provided with said orifice through which



the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser, and other means for heating the other part of said chamber during the discharge of the liquid metal to the ingot mould and the filling of said mould with the liquid metal.

9. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, means to maintain the upper part of said chamber provided with said orifice through which the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser, the other part of said chamber being heat insulated during the discharge of the liquid metal to the ingot mould and the filling of said mould with the liquid metal, and cooling means adapted to be substituted for the heat insulating means of the ingot mould chamber after the filling of the ingot mould and before it is taken out of the chamber.

10. Apparatus for the production of a metal by condensation of its vapors which comprises a reaction chamber for the heat treatment of the ore under vacuum, a condenser comprising two successive zones communicating one with the other, the first zone communicating with the reaction chamber and the second zone with a vacuum pump, means to establish and maintain in said zones respectively temperature and pressure conditions adapted to cause the metal vapors to condense in the first zone to the liquid state and in the second zone to the solid state, the communication between the two zones being arranged to create in the vapors current sucked by the vacuum pump a loss of pressure capable of ensuring the required difference of pressure between the first zone and the second zone, a tight chamber in which is placed an ingot mould to receive the metal, said chamber communicating by an orifice through its upper part with the lower part of the first zone of the condenser in which the metal vapors condense to the liquid state, means to maintain the upper part of said chamber provided with said orifice through which the metal is discharged to the ingot mould at the same temperature as the first zone of the condenser, other means for heating the other part of said chamber during the discharge of the liquid metal to the ingot mould and the filling of said mould with the liquid metal, and cooling means adapted to be substituted for the heating means of the ingot mould chamber after the filling of the ingot mould and before it is taken out of the chamber.

11. Condensing apparatus for the production

of magnesium or the like comprising a plurality of airtight chambers, means to deliver metal vapors to one of the chambers, means to evacuate the other of the chambers, means connecting the chambers of such size that a substantially constant difference in pressure exists within them when the evacuating means is in operation, and means to maintain the chambers at selected temperatures whereby part of the vapors may be condensed in one and part in the other chamber.

12. Condensing apparatus for the vapors of magnesium or the like comprising a chamber maintained under conditions of temperature and pressure adapted to condense the vapors of a metal to a liquid, and means to receive the condensate comprising an ingot mold containing and surrounded by oil.

13. Condensing apparatus for the condensation of the vapors of magnesium or the like comprising interconnected chambers, means for maintaining one of said chambers at a temperature and a pressure adapted to condense the metal vapors to a liquid, removable means to receive the condensed liquid, means to maintain the other of said chambers at a temperature and a pressure adapted to condense the vapors of the metal to a solid, and means to remove the solid-containing chamber.



GEORGES BASSEREAU.


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