Dec. 18, 1945. J. C. RADEMAKER 2,391,193

PROCESS FOR THE PRODUCTION OF MAGNESIUM

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Fig 1

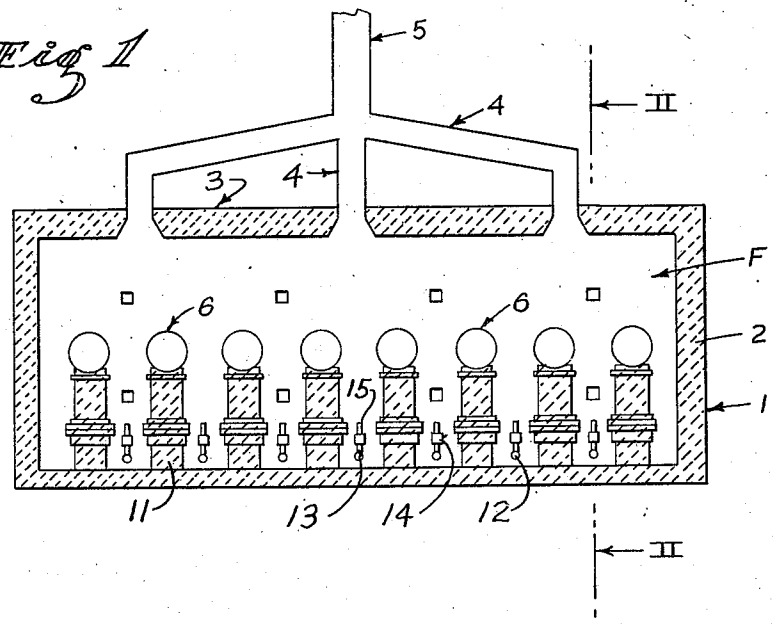
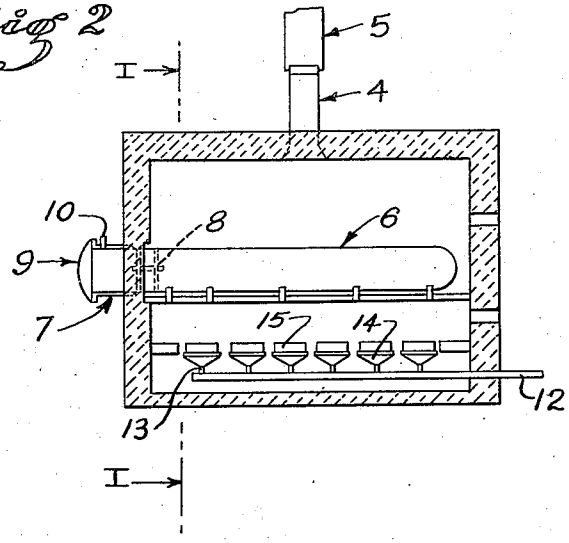


Fig 2



INVENTOR.
JOHN C. RADEMAKER
BY
Brown Jackson Boettcher Dienner
ATTY.

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PROCESS FOR THE PRODUCTION OF MAGNESIUM

John C. Rademaker, Palo Alto, Calif., assignor, by mesne assignments, to The Anglo California National Bank of San Francisco, San Francisco, Calif., a national banking association, as trustee for the benefit of Reconstruction Finance Corporation

Application August 4, 1944, Serial No. 548,027

2 Claims. (Cl. 75—67)

This invention relates to the production of metallic magnesium by the reduction of magnesium oxide-containing raw materials, particularly of calcined dolomite or magnesite, with the aid of a reducing agent such as silicon, ferrosilicon, the silicides of calcium and magnesium, aluminum, or mixtures thereof, or of similar reducing agents which do not generate gaseous products of oxidation and is a continuation-in-part of my co-pending application Serial Number 492,465 filed June 26, 1943.

It is well known that in effecting the above reduction operation with the use of a continuously fed furnace various difficulties are encountered as well in the type in which the operation is effected at a temperature above the melting point of the reaction components, as in the other type in which the mixture is heated at a temperature below its melting point. Both processes are disadvantageous. The reduction of magnesia in the liquid state is a rather slow reaction leaving behind a considerable proportion of the unaltered magnesia; moreover disengagement of the magnesium vapor from the molten metal mass is difficult; finally there is an appreciable attack on the furnace lining by the molten slag. On the other hand reduction exclusively in the solid state, while proceeding at a more rapid rate, gives rise to difficulties in conducting the operation such as to obtain economical rates of vapor flow, the vapor evolved will carry over dusty impurities into the condenser, as a consequence of which the recovered metal will not answer to practical purity requirements.

The art has therefore resorted to batch or intermittent processes. However, such operations have generally been disadvantageous for the reason that relatively small quantities of metal are recovered in the course of a single operation and because it has been considered useful to preheat the charge containing the mixture of magnesium oxide-containing raw material and the reducing agent. Furthermore, another great disadvantage of the prior art is that it has always been thought necessary to delay discharging the retort until it cools to a temperature below 80–100° C. since otherwise the magnesium would react with the atmospheric oxygen and nitrogen, which reaction may be so violent that the magnesium may be set on fire.

It is the object of the invention to enable the magnesium vapor to be disengaged at a rate high enough to obtain commercially economical rates of vapor flow. It is also an object of the invention to recover metallic magnesium in a very compact deposit, practically free from solids carried over by the vapor. Another object of the invention is to reduce the time required for carrying out the whole cycle of operations to a minimum and to thereby enable the operation to be carried out on a large scale. A further object of the invention is to provide apparatus for carrying out the process with efficiency and economy, and this application is directed to such apparatus.

Other objects and advantages will be apparent from the following specification in connection with the accompanying drawing which shows one form of apparatus for carrying out the invention.

Referring to the drawing:

Figure 1 is a horizontal elevation in section through the apparatus embodying my invention;

Figure 2 is a section taken on the line 2—2 of Figure 1.

Referring to Figures 1 and 2, a furnace F is shown which advantageously is provided with an outer shell or casing 1 having a refractory lining 2 which serves as the insulating medium for the walls and the flat top roof 3. The furnace roof has a plurality of outlets 4 leading to stack 5 which permit any products of combustion to pass from the furnace.

A series of retorts 6, open at one end and closed at the other, preferably of the metallic type, are horizontally disposed within the furnace in parallel relation to each other and in such manner that the end section of each retort extends outside the front wall of the furnace and forms a condenser section of a size sufficient to collect the crystalline metallic magnesium produced in a single cycle. A heat dam or reflector 8 is inserted within each retort in a position which separates the condenser section from that section of the retort exposed to the furnace. A dished lid or closure 9 is provided to seal each of the retorts 6 in an air-tight manner. Vacuum pipes 10 extend through the condenser section 7 into each retort. The height of the retorts 6 within the furnace should be sufficient to permit ready access to them either manually or by mechanical means, and furthermore, they are supported at such height by firebrick piers 11 which are corbeled or widened out in their mid-section (see Figure 1).

The furnace may advantageously be heated by natural gas which is supplied through a series of manifolds 12 and pipes 13, which lead to the burners 14. The burners 14 have one or more firebrick flamespreaders 15 fixed at their tops and

are located in series immediately adjacent the corbeled mid-sections of the piers slightly below the retorts and in parallel relation thereto. When the burners 14 are lit and the furnace becomes heated, the firebrick flamespreaders 15 become incandescent as well as the adjacent piers supporting the retorts, and as a consequence thereof there is no flame visible above the firebrick and the whole interior of the furnace above the piers 11 and the firebrick 15 serves as a muffle chamber. The corbeled sections of the piers 11 protect the gas manifolds and pipes located immediately below them from the heat of the incandescent zone. It is to be noted that the bricks supporting the retorts 6 at the top of the piers are preferably of the chrome type so that there will be no reaction with the retort itself.

It is to be understood that any type of heat dam may be employed in carrying out the invention so long as it satisfactorily separates the heated section of the retort containing the retort charge from the condensing section and at the same time permits the passage of the evolved magnesium vapors.

In operation the furnace and retorts are heated to normal reduction temperature, that is, to a temperature within the range of 1150 to 1260° C. and the retorts are charged with compacted mixtures of magnesium oxide-containing raw material and a reducing agent. Thereafter, the heat dam 8 is inserted within the retort in its proper position and the vacuum-tight lid 9 is inserted and the retort is sealed. A relatively low vacuum is applied to the retort through suitable pumps leading to pipe 10. This vacuum is advantageously within the range of from 10 to 150 millimeters, absolute pressure, or at least sufficiently low to prevent any magnesium from being distilled in the condenser section of the retort. The purpose of this low vacuum step is primarily to eliminate the necessity of preheating the furnace charge by removing concomitants such as water vapor, carbon dioxide, air, and dust. The low vacuum is maintained for a period sufficiently long to achieve the above end.

After the low vacuum period is over, additional vacuum pumps are put on the line leading to connection 10 and the vacuum within the retort is increased to a point where the reaction between the reducing agent and raw material takes place at commercial rates and magnesium vapor is rapidly evolved and condensed. At the temperatures prescribed hereinabove the magnesium is distilled when the pressure is lowered to within the range of from 10 to 150 microns, absolute pressure.

In passing from the retort to the condenser, the evolved magnesium vapors flow through the relatively small tubular passage provided in the center of the heat dam at a particularly high velocity and as a consequence thereof strike the walls of the condenser section with considerable force and form a condensed deposit of crystalline magnesium that is very solid and compact.

After the available magnesium contained in the retort charge is completely distilled and recovered as crystalline metallic magnesium in the condenser section the retort may be opened. Any sodium evolved during the course of the reaction may be collected in a suitable collector (not shown) which, if used, is advantageously located near the outer end of the condenser section and can be readily removed without igniting the magnesium. There is no reaction when the retort is opened and the magnesium is exposed to the oxygen and nitrogen of the atmosphere.

It is to be understood that the above cycle may be repeated without lowering the furnace temperature so long as the spent residue remaining in the retort is discharged. In fact the expedient of maintaining the retorts at substantially constant temperature throughout the entire cycle is advantageous because it prolongs the life of the retorts by preventing the occurrence of heat shocks and thermal stresses therein which normally occur upon substantial variations in temperature. Consequently, as soon as the retort residue from one cycle is discharged the retort may be recharged, sealed and the vacuum applied in the new cycle as previously described.

It has been found advantageous to use a quantity of reducing agent in the furnace charge slightly in excess of that necessary to reduce the available magnesium contained in the magnesium oxide-containing raw material. It is useful to recover the magnesium in large masses of solid compact form as distinguished from small pieces because the larger masses may be melted with less melting loss during the subsequent melting step. This manner of avoiding melting losses represents considerable saving in large scale operations.

It is desirable to prepare the feed for the retort charge in a very compact form comprising a mixture of the raw material and the reducing agent for the reason that the presence of dust decreases the purity of the magnesium metal recovered and cuts down heating efficiency within the retort. The compacted material may be in the shape of nodules, pellets, briquettes or the like, which may be of any suitable size so long as they do not present a dust problem.

In preparing the retort charge it has been found advisable to grind the materials before the compacting step. The desirable particle size for the raw material-containing magnesium oxide, for example dolomite, may be within the range of from 60 to 90 percent minus 200 mesh with very few particles coarser than minus 50 mesh. The particle size of the reducing agent, for example ferro-silicon, may be within the range of 50 to 75 percent minus 200 mesh with traces up to minus 10 mesh.

The following is an example of a typical manner of carrying out my invention:

Example

Briquettes of a volume of roughly about 1 cubic inch and comprising 4.5 parts of dolomite ground to about 75 percent minus 200 mesh and 1 part ferro-silicon ground to about 65 percent minus 200 mesh are charged to a retort heated to a temperature of about 1165° C. The retort is sealed and a vacuum of about 60 millimeters, absolute pressure, is applied for a period of about one and one-half hours to remove all concomitants from the charge and the retort itself. Thereafter the vacuum is increased to about 50 microns, absolute pressure, and magnesium vapor is evolved from the retort charge and condensed in the cool section. The high vacuum is maintained for a period of about six and one-half hours and immediately thereafter the vacuum is broken with air and the retort opened. Under the conditions of temperature, pressure, and ratio of feed prescribed above, it is found that the available magnesium present in the retort charge is completely

recovered as solid crystalline magnesium metal of high purity.

It is to be understood that any type of retort may be employed in carrying out my invention so long as it is capable of withstanding the conditions to which it is subjected during the course of the process. Retorts with a single open end, or with each end open and extending beyond the furnace walls may be utilized. The retorts may be made of any type of heat resistant material such as high temperature resistant metals and refractories. A particularly useful type of retort which has been found to be of considerable value from the standpoint of economics and ease of manufacture comprises a retort of mild steel having an outer coating of alloy steel. The mild steel base material may vary in thickness from ¾ to 2 inch and the coating of alloy steel may vary in thickness from ⅛ to ⅜ inch. The inside diameters of satisfactory types of retorts vary from 8 to 12 inches. The welded coating on the outside of the mild steel retort may be applied automatically or by hand welding. This coating may advantageously be alloy steel of the type having a chromium content of 26 to 30 parts and a nickel content of 14 to 20. The higher chrome content is preferable.

I claim:

1. In the production of metallic magnesium by reduction of magnesium oxide-containing raw material with the aid of reducing agents generating non-gaseous products of oxidation, the method which comprises forming a reaction mixture comprising the initial material and a reducing agent, heating said mixture under vacuum at normal reduction temperatures for a period sufficient to completely remove undesirable concomitants without distilling magnesium, thereafter increasing said vacuum while maintaining constant temperature to cause magnesium vapor to be given off, and cooling said vapor to a temperature below the solidification point of magnesium.

2. The process of claim 1 wherein the reducing agent is ferrosilicon.

JOHN C. RADEMAKER.