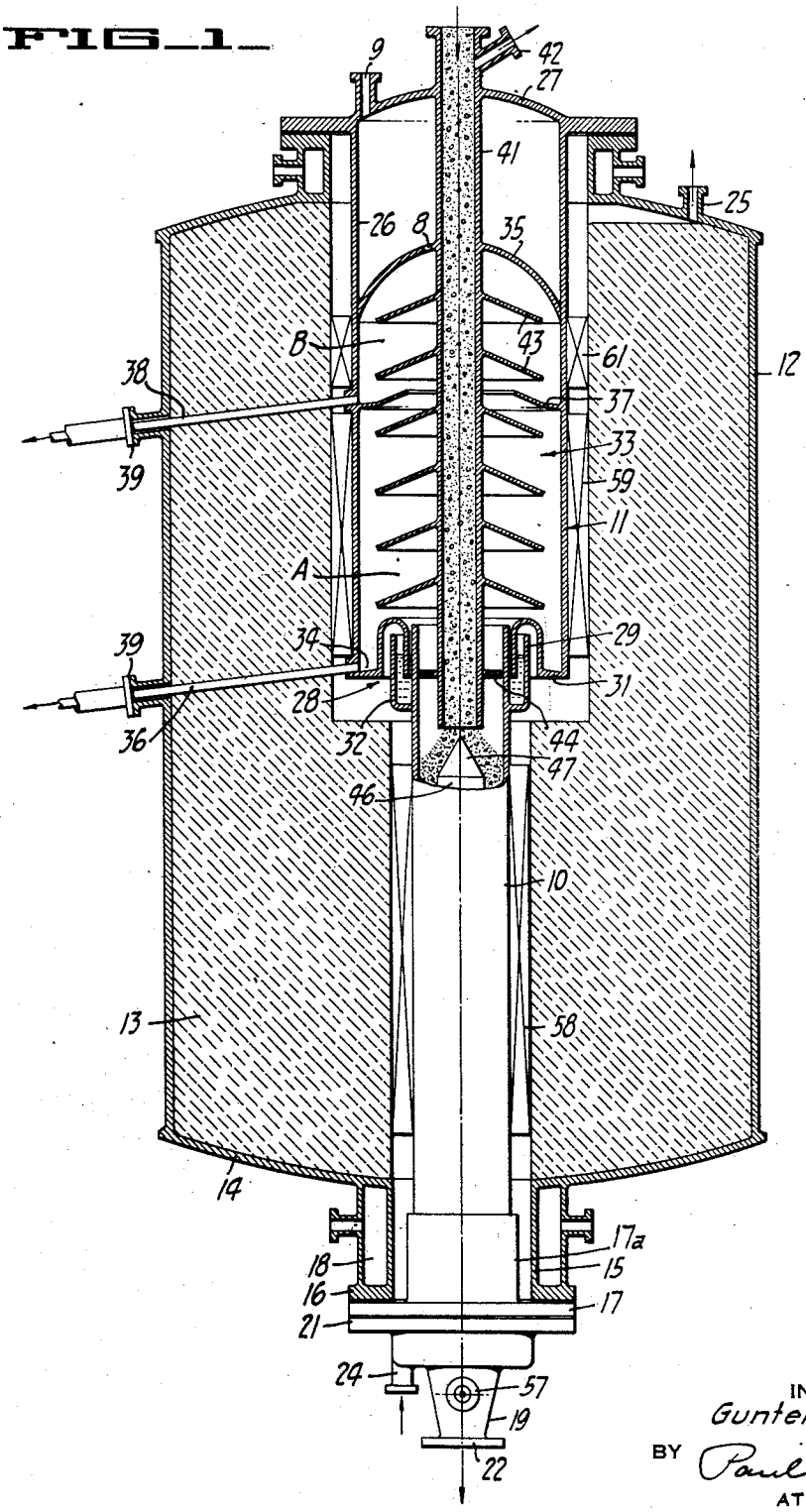
Dec. 21, 1943. G. H. GLOSS 2,337,042

APPARATUS AND METHOD FOR MANUFACTURE OF MAGNESIUM METAL

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FIG. 1



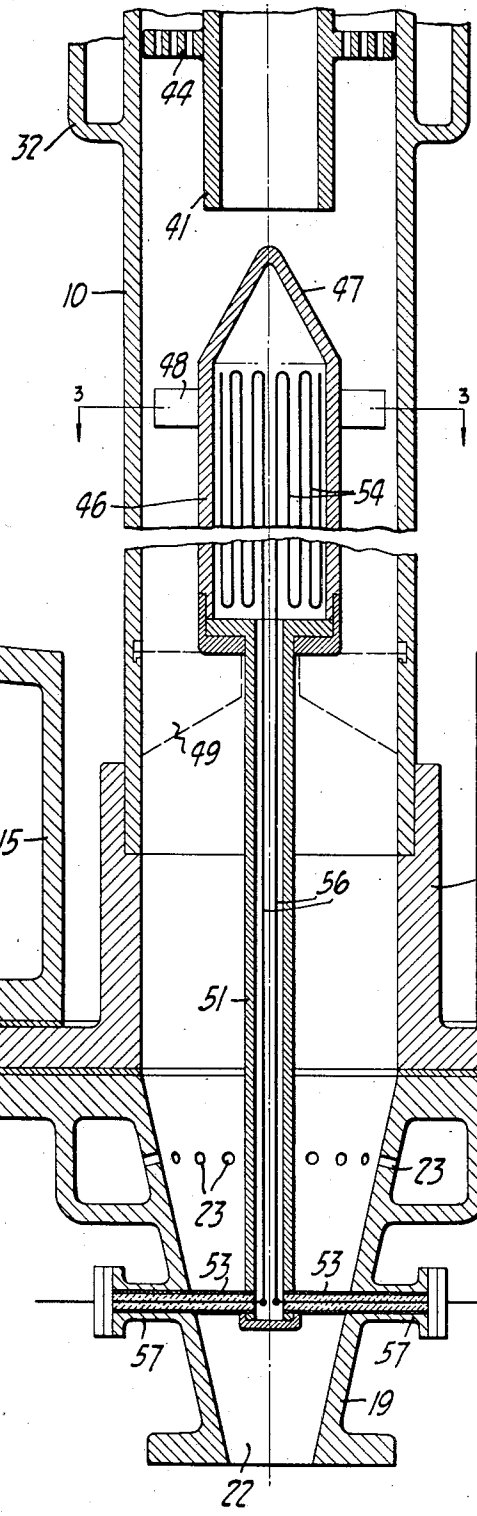
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APPARATUS AND METHOD FOR MANUFACTURE OF MAGNESIUM METAL

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FIG. 2.



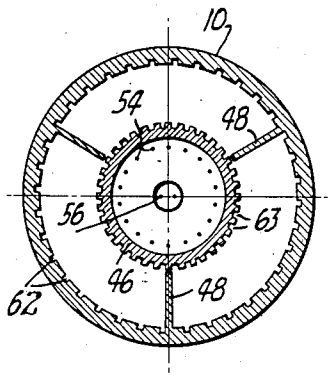


FIG. 3.

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APPARATUS AND METHOD FOR MANUFACTURE OF MAGNESIUM METAL

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FIG 4

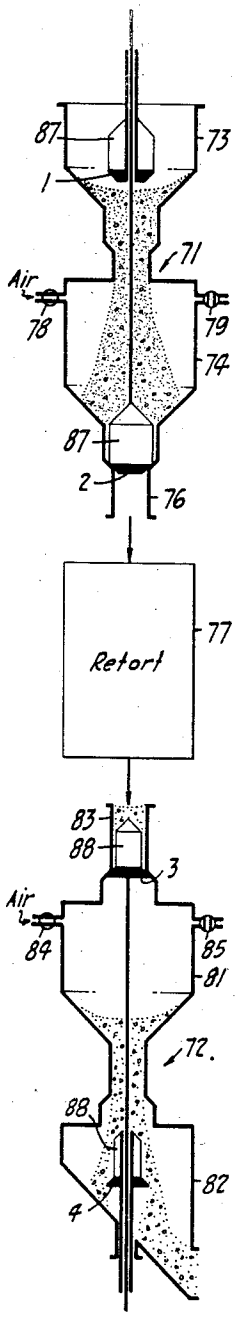


FIG 5

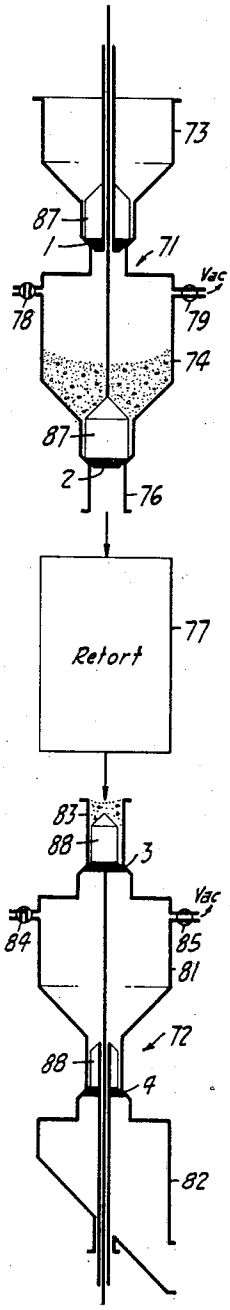


FIG 6

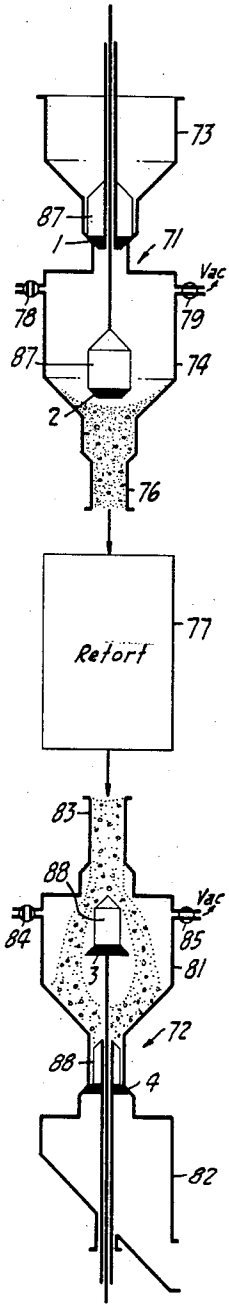
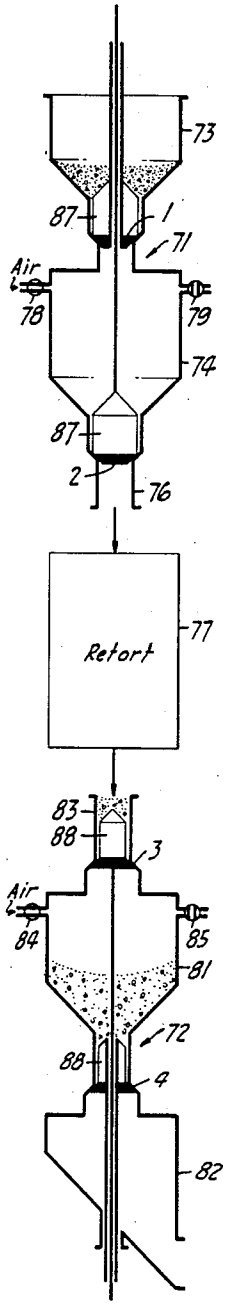


FIG 7



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UNITED STATES PATENT OFFICE

2,337,042

APPARATUS AND METHOD FOR MANUFACTURE OF MAGNESIUM METAL

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Application November 4, 1941, Serial No. 417,796

11 Claims. (Cl. 75—67)

This invention relates generally to apparatus for the manufacture of metallic magnesium and to methods for use in connection with the same. More particularly, the invention relates to apparatus and methods in which evolved magnesium vapor is condensed, and it is applicable for manufacture of magnesium by thermal reduction of magnesium compounds like magnesium oxide.

In the thermal reduction of magnesium oxide with reducing agents like ferrosilicon or calcium carbide, it is customary to form the reacting ingredients into pellets or briquets, which are then heated to effect a reducing reaction to release magnesium vapor. If the metal is condensed in liquid form, it is desirable to carry out the reaction under reduced pressure, such as from 10 to 150 mm. of mercury, which necessitates the use of sealed equipment maintained under partial vacuum.

It is an object of the present invention to provide equipment of the above character which is capable of continuous operation for large scale production of magnesium metal.

An additional object of the invention is to provide apparatus in which the retort is capable of expansion and contraction, without imposing undue stresses on certain parts and without introducing difficulties with respect to maintenance of the desired vacuum.

A further object of the invention is to provide improved provision for flow of evolved magnesium vapor from various parts of the charge, to the condensing chamber.

Another object of the invention is to provide improved means for applying heat to the charge, which will provide for faster transfer of heat and better heat distribution.

Another object of the invention is to provide a novel method for carrying out a thermal reduction operation for manufacture of magnesium, and which method is particularly adapted for use in conjunction with the present apparatus.

An additional object is to provide for separate withdrawal of sodium which may be formed simultaneously with magnesium.

Additional objects and features of the invention will appear from the following description in which the preferred embodiment has been set forth in detail in conjunction with the accompanying drawings.

Referring to the drawings:

Figure 1 is a side elevational view, in cross section, illustrating apparatus incorporating the present invention;

Figure 2 is an enlarged sectional view of a portion of the apparatus shown in Figure 1;

Figure 3 is a cross sectional detail taken along the line 3—3 of Figure 2;

Figure 4 is a side elevational view, diagrammatically illustrating suitable vacuum locks or valves for charging and for removing reacted residue from the apparatus of Figure 1; and

Figures 5, 6 and 7 are figures similar to Figure 4, but showing the valves in different operating positions.

The apparatus illustrated in the drawings consists of a tubular reort 10, which is preferably cylindrical in form, and which is disposed vertically. Various materials can be utilized for the construction of the retort. For example, in some instances it may be made of a metal or metal alloy capable of successfully withstanding the temperatures to which it may be subjected. However, in conjunction with the treatment of briquets utilizing calcium carbide or ferrosilicon as reducing agents, I prefer to use a refractory, such as graphite, fused alumina or sillimanite. A tube made of such refractory material can be formed either as a single molded or cast piece, or it can be fabricated by cementing together suitable refractory segments or rings.

The upper opened end of the retort 10 communicates with a condenser designated generally at 11. Both the retort and the condenser 11 are enclosed within a suitable sealed metal housing 12. The interior of this housing is lined with a suitable refractory insulation 13, such as a magnesia brick.

To provide suitable means for mounting the retort 10, the bottom end wall 14 of the housing 12 is provided with an extension 15, which surrounds the lower part of the tubular retort, and which is provided with a flange 16. The lower end of the retort tube 11 is also provided with a flange 17, which is clamped to and sealed with respect to the flange 16. Flange 17 is welded to a collar 17a, which in turn is sealed, cemented or threaded on the lower end of the refractory retort 10. Extension 15 is shown being cooled by the water jacket 18. Below the flange 17 there is a restricted discharge nozzle 19, which also has a flange 21 for forming a sealed clamping engagement with respect to flange 17. The discharge opening 22 of nozzle 19 connects with suitable equipment (hereinafter described) having vacuum locks or valves for removing the spent charge without releasing the vacuum.

For a purpose to be presently explained, nozzle

19 is shown provided with small openings 23 through which small amounts of an inert gas like hydrogen or hydrocarbon gas can be introduced from a source of supply connected with pipe 24. Housing 12 is normally connected to evacuating equipment through pipe 25. A partial pressure is maintained in the housing slightly greater than that maintained in the retort and condenser.

The condensing means 11 consists of a cylindrical shaped shell 26, the upper end of which is secured to the cover plate 27 of the housing 12. An expansible and liquid sealed coupling 28 serves to connect the lower end of shell 26 with the upper end of the retort 10. This coupling can be conveniently formed of a downturned annular skirt 29, formed as a part of the bottom wall 31, and which is loosely accommodated within the annular trough 32 formed about the upper end of the retort 10. Trough 32 contains a suitable sealing medium which is liquid at the temperatures of operation, such as lead.

The inner condensing chamber 33, which receives magnesium vapor from the upper end of the retort 10, is divided into two portions or zones A and B. Zone A is for condensing released magnesium vapor. Zone B is for condensing any sodium vapor which may be released simultaneously with magnesium. The upper end of zone B is defined by the dome shaped wall 35. Sodium vapor may be encountered particularly in the treatment of briquets consisting of magnesium oxide and calcium carbide, together with a sodium compound, such as sodium chloride. Vent 8 together with pipe 9 can serve to connect the interior of the condenser to a source of vacuum.

The condensed liquid magnesium is collected within the annular cup-like space 34, underlying the condensing zone A, and is withdrawn through pipe 36. Another annular cup 37 underlies the zone B, and connects with the sodium removal pipe 38. Pipes 36 and 38 extend to the exterior through suitable sealed glands 39, and connect with suitable equipment such as known to those skilled in the art for receiving the liquid metal without, however, releasing the vacuum within the apparatus. For example, the means employed can be in the form of an oil chamber in which the liquid metal is solidified in granular form in a countercurrent flow of hydrocarbon oil, granulated metal and some oil being intermittently discharged through a lock chamber or double valve. The oil is continuously circulated through a filter, cooled and returned to the chamber, or the metal may be cast directly into interchangeable molds which can be evacuated while connected with the discharge pipes 36 and 38. Pipes 36 and 38 must be suitably heated to avoid solidification of the metals.

The briquets being treated are supplied to the retort 10 through the vertical feed tube 41. This tube extends down through the cover plate 27, axially of the condenser 11, and has its lower end terminating shortly below the upper open end of the retort 10.

The upper end of tube 41 connects with suitable equipment provided with vacuum valves or locks (hereinafter described), whereby briquets are supplied to the tube 41 at a rate commensurate with the capacity desired, without releasing the vacuum. A small pipe 42 is shown connected to the upper portion of tube 41, for the purpose of withdrawing certain gaseous products evolved while the charge is being preheated. Within the condenser 11, conical shaped metal baffles 43 are mounted upon tube 41, and serve as surfaces upon which the metal vapors condense, and as means for transmitting heat from the metal vapors to the tube 41, and to the material within the tube.

Attached to the lower end of the tube 41 and between this tube and the adjacent upper end portion of the retort 10 there is a grill or baffle 44 which prevents solid particles arising with the metal vapors from entering the condenser without, however, preventing passage of evolved vapor.

Extending axially through the tubular retort 10, there is an inner tubular core 46, having a conical shaped upper end portion 47 disposed below the lower open end of tube 41. Webs 48 and 49 extending inwardly from the walls of retort 10 serve to retain the tubular core 46 centralized. Webs 49 are removable and also serve to support the core. The lower end of core 46 is coupled to an extension tube 51, which in turn has its lower end attached to the laterally extending pipes 53. Electrical resistor elements 54 disposed within tubular core 46 form a heating unit connected to the conductors 56. These conductors extend downwardly through tube 51, and outwardly through the lateral pipes 53 and the gas-tight fittings 57. Tubular core 46, together with tube 51, can be formed of a suitable material capable of withstanding the temperatures to which it is subjected, such as a suitable refractory the same as retort 10.

In addition to the electrical heating units provided within the tubular core 46, it is desirable to provide an electrical heating unit 58 about the retort 10. In addition to the heating units described, one or more additional heating units 59 and 61 are shown surrounding the shell 26 of the condenser. These heating units serve to maintain the walls of the condenser well above the fusion points of magnesium and sodium. Preferably, a proper temperature gradient is maintained between zones A and B so that the walls surrounding zone A are maintained at a temperature below the vaporization point of magnesium, but above that of sodium, and with the walls surrounding zone B maintained at a temperature below the vaporization point of sodium. Where a relatively high condensing rate is being maintained, the upper or all of the condensing chamber may require controlled cooling to maintain the desired temperatures. Such cooling can be carried out by use of cooling coils through which fluids such as oil or glycerin are circulated.

To facilitate evolution of magnesium vapor from the charge of material undergoing treatment and the flow of this vapor to the condenser, I provide a plurality of upwardly extending ducts adjacent both the outer and inner portions of the charge. Thus, as shown in Figure 3, the inner periphery of the retort 10 is provided with a plurality of circumferentially spaced and upwardly extending grooves or passages 62. It is desirable to also provide the outer periphery of the tubular core 46 with circumferentially spaced and vertically extending grooves 63. These grooves should be of a width substantially smaller than the briquets or fragments of the charge of material undergoing treatment, so that at all times they provide unobstructed ducts for flow of vapor.

To describe operation of the apparatus, and the carrying out of the present method, it will be presumed that the briquets being subjected to thermal reduction consist of a homogeneous

mixture of magnesium oxide with calcium carbide. The heating units 54 and 58 are supplied with current to transfer the required amount of heat through the walls of the tubular core 46, and through the walls of the retort 10, so that the briquets introduced into this tube are heated to an elevated temperature, such as of the order of from 1,100 to 1,400° C. The equipment is maintained under a reduced pressure such as from 10 to 150 mm. of mercury. An adequate supply of briquets is sent to the upper end of the tube 41 and as such briquets are removed through the lower opening 22, the column of briquets within retort 10 progresses downwardly, and is continually maintained by fresh briquets delivered from the lower end of tube 41. Preferably, this progression of briquets is by periodic increments, controlled by periodic removal of quantities of spent briquets through opening 22. The quantities regularly removed in this fashion are preferably relatively small compared to the total mass of briquets undergoing treatment in the retort 10. Within the retort, the briquets are heated to an elevated temperature in a region surrounding the tubular core 46, to complete the desired reducing action with release of magnesium vapor. The released vapor can pass unwardly through the grooves 62 and 63 to the condenser. Within the condenser, the magnesium vapor condenses upon the baffles 43, and the condensed metal is collected as a liquid in cup 34, and withdrawn through pipe 36. The heat thus absorbed by baffles 43 is conducted to the feed tube 41, and is transmitted to the column of material passing through this tube. Thus, material progressing through feed tube 41 is being preheated so that by the time it enters the retort 10, it is already at an elevated temperature, such as of the order of 800° C.

Preheating of the briquets as described above is highly desirable, because it serves to prevent undesirable release of certain gases within the retort.

Irrespective of measures which may be taken to minimize presence of moisture in the briquets, the briquets will have a small amount of moisture content before they are supplied to the apparatus. Furthermore, they may contain small amounts of combined carbon dioxide. If such briquets were immediately placed within the retort and heated to the reaction temperature, released water vapor and carbon dioxide would interfere with the desired efficiency of the reaction and with the purity of the metal produced. Moisture has been found to cause formation of hydrocarbon gases, because of reaction with calcium carbide. Gases released during the preheating operation, as the briquets are passed through the feed tube 41, are substantially entirely removed by the vacuum equipment applied to pipe 42, so that such gases do not enter the retort or the condenser. Evacuating equipment applied to 9 serves to maintain a high vacuum in the condenser and in the upper portion of the retort. By regulating evacuation through 42 a slightly higher pressure is maintained in tube 41 than in the condenser and upper part of the retort to prevent magnesium vapors from entering this tube.

The charge of briquets within the retort 10 is progressed downwardly at such rate that by the time a given part of a charge reaches the lower end of the tubular core 46, the reaction is substantially complete. While the charge is progressing from this point to the discharge opening 22, it is being cooled to a relatively lower temperature, such as a temperature of the order of 100 to 200° C. Small amounts of an inert gas like hydrogen or hydrogen gas are bled into the pipe 24, and through openings 23 for the purpose of maintaining the proper pressure and aiding the upward flow of evolved magnesium vapors. Also, gas introduced in this manner aids in cooling the reaction residue in the lower part of the retort tube.

A feature of the apparatus described above is that the tubular retort 10 may expand and contract with respect to condenser 11, without placing any undue strains upon its walls, or upon other vacum type joints or walls of the housing 12. A liquid seal coupling 28 permits both lateral and longitudinal movements of the upper part of the retort, without in any way interfering with the desired seal in the connection between the retort and the condenser. In addition, the inner tubular core 46 may expand and contract independently of the retort tube 10. These characteristics are particularly desirable where the retort and also the inner tubular core 46 are formed of refractory materials such as graphite, fused alumina, or sillimanite. Repairs or replacements are facilitated because the retort 10 and cup 32 can be removed through the bottom of the housing. In this conection the heating element 58 should be constructed to either pass cup 32, or the element should be removable to permit cup 32 to pass down through the passage in the refractory lining 13.

My apparatus and method make possible large scale and low cost production of magnesium metal, because equipment can be constructed to relatively large sizes for handling large amounts of material. The apparatus and method are also capable of relatively efficient yields, particularly because of the manner in which the briquets are preheated before they are introduced into the retort. Where the briquets contain magnesium oxide, calcium carbide and a sodium compound such as sodium chloride, the sodium metal is separately condensed and removed to pipe 38, thus avoiding contamination of the main body of magnesium metal, and at the same time providing a valuable by-product.

The heat is relatively rapidly and uniformly distributed to the charge of material undergoing treatment, particularly because a large percentage of the heat required is supplied from the interior of the charge. Evolved magnesium vapor need not pass for considerable distances through the charge, because such vapors can readily pass through the upwardly extending ducts formed by the grooves 62 and 63 to the condenser.

Figures 4 to 7 inclusive illustrate suitable auxiliary equipment for use with the apparatus of Figure 1, and by means of which the briquets are supplied to the feed tube 41, and are removed from the lower end of the retort tube, in regular synchronized intervals, without interfering with maintenance of the desired partial vacuum. In general, this auxiliary equipment includes the charging unit 71, and the discharging unit 72. The charging unit 71 includes a hopper 73 into which the briquets are supplied, and which is at atmospheric pressure, together with a vacuum chamber 74 into which the briquets are discharged from hopper 73. Chamber 74 discharges briquets into the tube or conduit 76, which in turn connects with the retort 77. Reciprocating

valves 1 and 2 serve to close off the passage connecting hopper 73 with chamber 74, and chamber 74 with the outlet 76. Valves 78 and 49 also serve to connect chamber 74 to the atmosphere and to a source of vacuum, respectively.

The discharge unit 72 similarly includes a vacuum chamber 81, and a discharge hopper 82. Conduit 83 connects with the discharge end of the retort 77, and communication between this conduit and chamber 81 is controlled by a valve 3. Valve 4 serves to control communication between chamber 81 and discharge hopper 82. Valves 84 and 85 control communication between chamber 81 and the atmosphere and a source of vacuum, respectively. Valves 1 to 4 inclusive and also valves 78, 79, 84 and 85 are connected to suitable operating mechanisms whereby they are operated automatically and in a predetermined sequence. Also valves 1 to 4 inclusive are shown provided with piston-like extensions 87 and 88 to facilitate movement to closed positions without crushing briquets.

A proper cycle of operation for the various valves has been illustrated in Figures 4 to 7 inclusive. In Figure 4 valve 2 is closed, while valve 1 is open to permit briquets to pass into the chamber 74. At the same time, valve 3 for unit 72 is closed, and valve 4 is open to permit residue in chamber 81 to discharge into hopper 82. Both valves 79 and 85 are closed, while valves 78 and 84 are open to the atmosphere.

After all of the material in hopper 73 has passed into chamber 74, and all of the residue in chamber 81 has been discharged to hopper 82, valves 1 and 4 are moved to closed position, as shown in Figure 5, and at the same time valves 78 and 84 are closed, and valves 79 and 85 are opened. Thus, chambers 74 and 81 are evacuated. As shown in Figure 6, valves 2 and 3 are now opened, thus permitting the briquets to pass from chamber 74 into the retort, and permitting residue to discharge into the chamber 81. This is accomplished without release of vacuum upon the retort.

After all of the briquets in chamber 74 have entered the retort, and after an equivalent amount of residue has been withdrawn from the retort past the open valve 3, valves 2 and 3 are again closed, as shown in Figure 7. Valves 79 and 85 are then again closed as indicated in Figure 7, and valves 78 and 84 are opened to permit venting to the atmosphere. Thereafter, valves 1 and 4 are again operated as illustrated in Figure 4. In the operation of the auxiliary equipment illustrated in Figures 4 to 7 inclusive, it will be evident that suitable means must be provided for supplying suitable amounts of briquets at regular intervals, to the feed hopper 73. Likewise, the capacity of this auxiliary equipment must be such that the batches of briquets introduced into the retort constitute a minor fraction of the briquets undergoing treatment. Thus, with automatic and continual operation of the units 71 and 72, the feeding and discharge of material to and from the retort will be substantially continuous.

In the foregoing particular reference has been made to the use of briquets containing magnesium oxide and a reducing agent. It is also possible to employ briquets containing impure magnesium particles for the purpose of refining the metal. Thus, the briquets can be formed from impure magnesium powder resulting from reduction of magnesium oxide with carbon, followed by rapid chilling of the evolved vapor (carbothermic process). The powder thus produced which contains some carbon and other impurities can be mixed with a suitable binder, and this mixture compressed into suitable tablets or briquets for charging the apparatus. When using the apparatus for this purpose, the temperature of operation of the retort is reduced to a value such as about 700° C., and therefore, if desired, the retort can be made of metal such as mild steel. The residue after evaporation of the magnesium metal is a powder which can be removed at a proper controlled rate by vacuum locks as previously described.

Another procedure which can be followed to advantage in the refining of impure magnesium powder is to mix the powder with a binder, such as a heavy petroleum oil, or like hydrocarbon. This mixture can then be extruded into fragments of suitable size and employed to charge the apparatus. In passing down through tube 41, preheating of such a charge will result in volatilization and cracking of the hydrocarbon, leaving only a carbon residue as the fragments reach the retort 10. Released hydrocarbon vapors are withdrawn upwardly through pipe 41.

This application is a continuation in part of my copending application Serial No. 398,946, filed June 20, 1941.

I claim:

1. In apparatus for the manufacture of magnesium metal by thermal treatment of briquets containing material capable of evolving magnesium vapor, a retort adapted to be heated, a condensing chamber, and a feed tube for introducing briquets into the retort, said feed tube extending through the condensing chamber and adapted to be heated by condensing magnesium vapor.

2. In apparatus for the manufacture of magnesium metal by thermal treatment of briquets containing material capable of evolving magnesium vapor, a retort adapted to be heated, a condensing chamber communicating with the retort for condensing evolved magnesium vapor, and a feed tube serving to supply briquets to the retort, said feed tube extending through the condensing chamber, and heat conducting baffles mounted upon the feed tube and serving to condense evolved magnesium vapor.

3. In apparatus for the manufacture of magnesium metal by thermal treatment of briquets containing material capable of evolving magnesium vapor, a vertically disposed tubular retort, the lower end of the retort having provision for withdrawing a spent charge of the briquets, means for heating the retort and a charge of briquets within the same, a condensing chamber extending above the upper end of the retort, a sealed expansible coupling serving to connect the upper end of the retort with the condensing chamber, a feed tube extending downwardly through the condensing chamber and serving as a passage for introducing briquets into the upper portion of the retort, and heat conducting baffles within the condensing chamber and mounted upon said feed tube.

4. In apparatus for the manufacture of magnesium metal by thermal treatment of material capable of evolving magnesium vapor, a tubular retort adapted to be heated, a condensing chamber communicating with the retort, means for maintaining a column of material to be treated within the retort, the inner walls of the retort being provided with grooves for conveying evolved

magnesium metal from the charge to the condenser.

5. In apparatus for the manufacture of magnesium metal by thermal treatment of material capable of evolving magnesium vapor, a vertically disposed tubular retort adapted to be heated, a condensing chamber communicating with the upper end of the retort, means for maintaining a vertical column of material to be treated within the retort, the inner walls of the retort being provided with a plurality of circumferentially spaced and vertically extending grooves for conveying evolved magnesium vapor from the charge to the condensing means.

6. In apparatus for the manufacture of magnesium metal by thermal treatment of material capable of evolving magnesium vapor, a tubular retort adapated to be heated and adapted to contain a column of material to be treated, a condensing chamber communicating with one end of the retort, a tubular core disposed within the retort, and heating means disposed within the core.

7. In apparatus for the manufacture of magnesium metal by thermal treatment of material capable of evolving magnesium vapor, a tubular retort adapted to be heated and adapted to contain a column of material to be treated, a condensing chamber communicating with one end of the retort, a tubular core extending within the retort and within the charge of material, the outer wall of the tubular core being provided with circumferentially spaced and longitudinally extending grooves for conveying magnesium vapor, and means within the tubular core for heating the same.

8. In apparatus for the manufacture of magnesium vapor by thermal treatment of briquets containing material capable of evolving magnesium vapor, a retort adapted to be heated and to contain a charge of the briquets, a gas tight condensing chamber for condensing evolved magnesium vapor, the interior of both the retort and the condenser being adapted to be evacuated, a liquid sealed expansible coupling serving to connect one end of the retort to the condenser, and a housing serving to seal off the space surrounding the coupling.

9. In apparatus for the manufacture of magnesium metal, a retort adapted to be heated and adapted to contain a charge of the material, the material when heated serving to evolve magnesium vapor, a condensing chamber for condensing evolved magnesium vapor, an expansible coupling serving to connect one end of the retort to the condensing chamber, said coupling being sealed with molten metal having a relatively low vapor pressure at a temperature corresponding to the vaporization temperature of magnesium, a housing serving to seal off the space surrounding the coupling, and means for evacuating the space surrounding the coupling, the interior of the retort, and the condensing chamber.

10. In a method for the thermal treatment of briquets containing material capable of evolving magnesium vapor, the steps of forming a downwardly progressing feed column of briquets of substantial height, causing briquets to be discharged from the lower end of said feed column to the upper end of a second column of briquets, causing the briquets in the second column to progress downwardly through a vertically extending treatment zone, heating the upper portion of the second column to effect release of magnesium vapor, absorbing heat from magnesium vapor evolved in the upper end of the column, and utilizing such absorbed heat for preheating briquets forming the feed column to prevent release of undesirable gases within the retort.

11. In apparatus for the manufacture of magnesium metal by thermal treatment of briquets containing material capable of evolving magnesium vapor, a vertically disposed tubular retort, a vapor condensing means communicating with the upper end of the retort, a tubular core extending axially within the retort, the outer wall of the tubular core being spaced from the walls of the retort, a vertically disposed feed tube extending above the retort, the lower end of the feed tube terminating within the upper end portion of the retort at a point adjacent thereto but spaced upwardly from the upper end of the core, means for supplying briquets to said feed tube whereby a column of briquets is maintained within the feed tube and whereby such briquets are fed from the lower end of the feed tube over the upper end of the core to maintain a column of briquets within the tubular retort, and means for supplying heat to said bore.

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