

July 4, 1950

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2,514,275

APPARATUS FOR CONDENSING METAL VAPORS

Filed Dec. 12, 1945

2 Sheets-Sheet 1

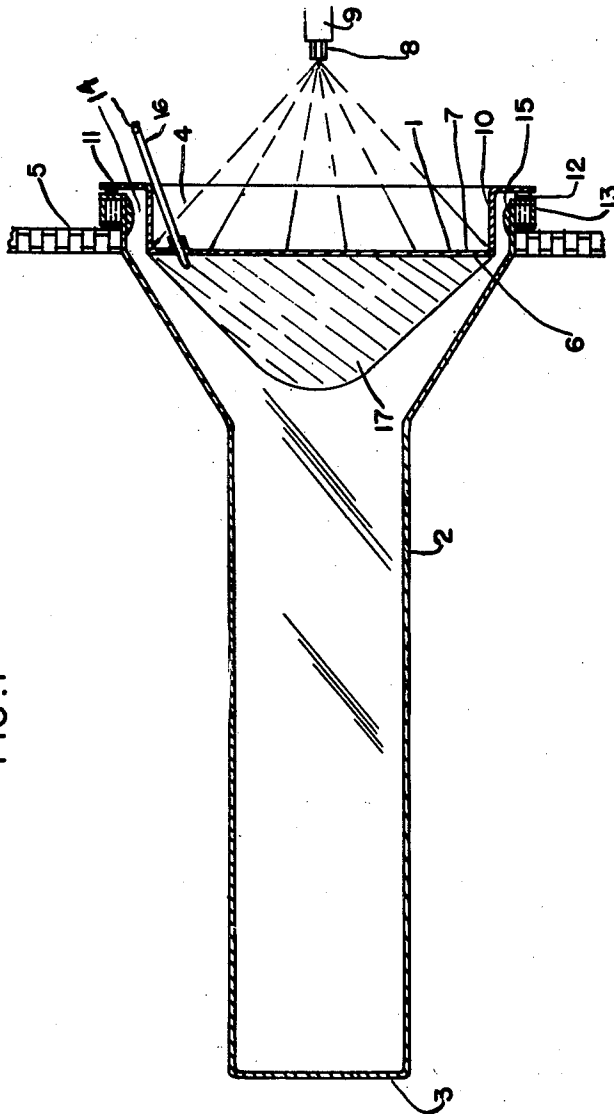


FIG. 1

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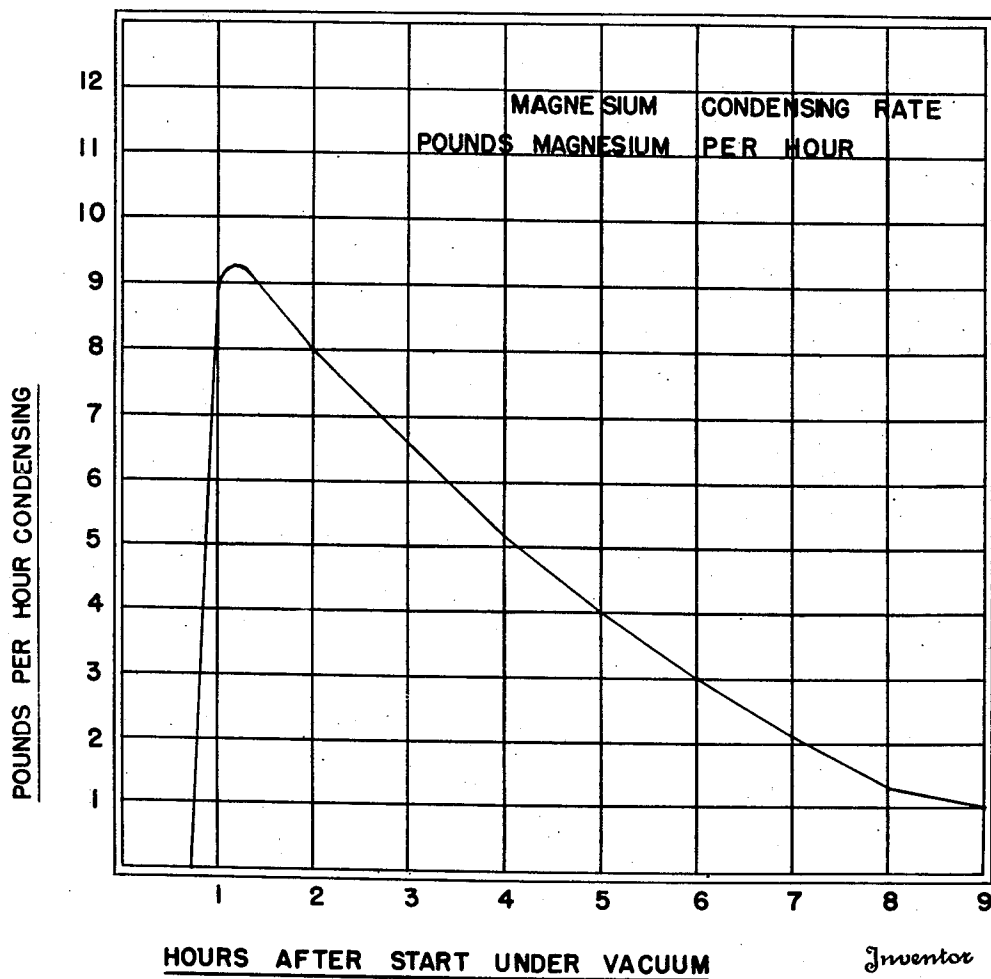
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2 Sheets-Sheet 2

FIG. 2



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2,514,275

APPARATUS FOR CONDENSING METAL
VAPORS

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America as represented by the Secretary of
the Navy

Application December 12, 1945, Serial No. 634,602

3 Claims. (Cl. 266—19)

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This invention relates to the condensation of vapors to the liquid or solid state, more particularly to the condensation of metal vapors under controlled conditions of temperature.

As is known, in the condensation of vapors to the liquid or solid state the control of the condensing temperature is an important factor, particularly in determining the physical characteristics of the condensate. Such temperature control is manifestly of paramount importance when the preferential or fractional condensation of a component of a mixture of vapors is desired.

The effective control of condensation temperature is of special importance in the condensation of magnesium vapors evolved in the thermal reduction of magnesium oxide or formed in a method of purifying secondary or scrap magnesium alloys by distillation or sublimation. The control of the condensing temperature of magnesium vapor to magnesium metal is important for two reasons. In the first place the temperature at which the condensation is effected to a very large degree determines the physical character of the condensed metal. If such condensing temperature is too low the metal condenses as a relatively loose crystalline structure; on the other hand if the condensing temperature is too high, i. e. above the melting point, the magnesium is condensed as a liquid. It has been found, as a practical matter, that the optimum type of deposit is obtained when the condensation temperature is slightly below the melting point. In these circumstances the metal is recovered as a dense or compact crystalline deposit. The physical characteristics of such deposits are of profound technical importance because the loss encountered in remelting the magnesium when alloying or casting into ingots is largely established by such physical structure. If the magnesium is in the form of loose fine crystals, losses of the order of 30% or higher may be encountered upon remelting; if the magnesium is in the form of open spicular crystalline structure a melting loss of the order of 10% or higher may obtain on remelting, while the desirable dense compacted deposits of magnesium may be liquified with a loss as low as 3%.

The control of the condensation temperature is important also because of the necessity of preferentially condensing the magnesium and the associated pyrophoric metals such as sodium and potassium. In current practice this is done by separately condensing such pyrophoric metals in a separate chamber or section of the condenser so that they are segregated from the magnesium and may thus be separately removed from the retort.

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Since such pyrophoric metals generally ignite spontaneously on exposure to the atmosphere they do not ignite the main deposit of magnesium. In this type of operation, the magnesium condensing temperature must be controlled such that the magnesium will condense while the vapors of the more volatile metals pass on in the retort for condensation in a separate section.

In the production of magnesium metal by condensation of magnesium vapors evolved from magnesium ores or from secondary or scrap magnesium alloys, the simplest condenser arrangement involves a metallic conducting surface which is cooled either by a water jacket or by air. However, such a simple type of condenser is not particularly effective. In operation it is substantially impossible to maintain such condensers at the desired elevated temperatures while insuring the abstraction of heat given up upon the condensation of the metal. The physical limits of such water or air cooled condensers are apparent. If a water cooled condenser is employed the surface in contact with the cooling water cannot be at a temperature above the boiling point of water otherwise steam will be formed. The formation of steam will automatically control the temperature of the contact surface at approximately 212° F. unless the system is under pressure. Maintaining a cooling system of this type under pressure is obviously undesirable. As pointed out previously since the desirable temperature of the condensing surface ranges from 600° F. to 1000° F., it is apparent that water cooled condensers will maintain the condensing surface at too low temperature for proper condensation. Air cooled condensers similarly have been proposed but have marked disadvantages. These condensers may be employed where the condensation rate is very low; however, as will be appreciated, since the heat capacity of air is quite small and since the heat given up by the condensation of magnesium vapor is of the order of 2500 B. t. u. per pound of magnesium, it is apparent that inordinate volumes of air are required for efficient cooling. For example, if an air cooled condenser is employed of a size sufficient to condense twenty pounds of magnesium per hour many thousands of cubic feet of cooling air would be required. This would involve the undesirable concomitants of very high velocities and expensive apparatus. For these reasons air cooling has been found to be impractical where reasonably high condensation rates are desired.

At the present time the condenser which has been generally employed, consists of a removable

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sleeve positioned in one end of the reduction retort and inside a water cooled jacket. Since the magnesium vapor, in such a unit, is condensed in a vacuum the space between the sleeve and the jacket forms, in effect, a gap or barrier through which heat from the sleeve may pass substantially only by radiation. Such radiation from the sleeve is relatively low at low temperatures but increases rapidly as the temperature of the sleeve increases. As is known, this transmission of heat by radiation varies substantially as the fourth power of the absolute temperature. Such prior type of condensers have functioned well in practice within a certain range of condensation rates. The temperature of the water jacket may be varied from room temperature to the boiling point of water without any great effect upon the physical structure of the condensate. This is for the reason that the heat radiated from the sleeve to the jacket is almost entirely dependent on the temperature of the sleeve and very little affected by the temperature of the jacket in the range of temperatures encountered. Such type of condenser, however, operates effectively only within a definite range of condensation rates. If too high a condensation rate is attempted to be achieved the condenser tends to overheat and some of the magnesium vapor condenses out as a liquid. In a system of this type which is designed for the condensation and removal of a solid such formation of a liquid metal may cause serious difficulty.

It has now been found that a very simple type of condenser may be provided which is effective over practically any desired temperature range and over a wide range of condensation rates. In its broadest aspects the improved method involves the control of the condensation temperature by utilizing a coolant which is effective to abstract heat at any desired rate to thus maintain the condenser temperature at the desired optimum degree for widely variable condensation rates.

In the drawings, Fig. 1 is an illustrative simplified physical embodiment of a retort and novel condenser, and

Fig. 2 is a graph showing the condensation rate for a typical cycle.

Broadly considered, the invention comprises the association of a condenser plate or surface with a magnesium distillation retort, on one surface of which plate magnesium is directly condensed and the other surface of which plate is contacted with a coolant having a desirably high heat abstraction. The flow of the coolant is adapted to be controlled to regulate the quantity of heat abstracted, during a given cycle, within closely controlled limits.

In the preferred embodiment of the invention, a water spray is used as a cooling medium. The temperature of the condensing material is thus simply controlled by varying the amount of water spray played upon the cooling surface. The water which impinges upon the cooling surface is instantly converted to steam and effective use is thus made of the high heat of vaporization of water. With such a system it is apparent that any temperature in the condensing material may be established and maintained by adjusting the water spray so that the proper amount of heat is abstracted from the condenser system. It is similarly apparent that with such a control the desired temperature may be maintained over widely varying condensation rates. In actual practice it has been found that with the described system

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the condensing temperature could be controlled precisely within a relatively few degrees between a range of from 300° F. to 1000° F. at various condensation rates.

The operation and efficacy of the improved method can be more readily appreciated from a consideration of the structure and function of a physical embodiment the essential features of which are diagrammatically illustrated in the single figure of the accompanying drawings.

As shown in the drawings, the condenser 1 is associated with a magnesium reduction retort 2. The retort may comprise an elongated cylinder formed with the closed end 3 and the open end 4. The retort may be of any desired size and in a typical construction may be about 10 feet long. The major portion of the length of the retort comprises a reduction and distillation zone and extends within a suitably fired furnace 5 for any desired distance. This reduction section may be about 10 inches in internal diameter. The remaining portion of the retort, which serves as the condensing zone, preferably is of increased diameter, as compared to the reduction zone. This condenser section of the retort may be of tubular configuration, or, as shown, may be of general frusto-conical shape. If desired, the zone may be segregated, as is done in the art, by means of a removable radiation shield which functions to protect the condensing zone from the radiant heat of the incandescent charge in the reduction zone.

The condenser unit 1 comprises a plate of high thermal conductivity, preferably of metal, on the internal surface 6 of which the magnesium vapors condense and on the external surface of which a spray of a liquid coolant is projected. The preferred cooling medium is water and may be applied by means of the spray device 8 connected with the fluid line 9. The spray unit may be adjusted to give any desired spray pattern and is provided with valves (not shown) to control the volume of the water applied to the exterior face 7 of the plate thereby controlling the condensation temperature. Preferably, the spray device is operated by compressed air, in the manner well known in the art.

The retort may be provided with a conduit connected to a source of vacuum so that the retort may be evacuated to any desired degree. Such vacuum line may be connected to the retort adjacent the open end 4 in any suitable manner.

The condenser plate 1, as shown, may form part of the closure for the retort. As illustrated in the drawings, the condenser plate proper is of a diameter considerably less than the internal diameter of the contiguous section of the retort. The condenser plate is formed with the lateral flange 10 spaced from the adjacent wall of the retort and the terminal vertical flange 11. As shown, the flange 11 abuts the gasket 12 and is adapted to be detachably secured to the retort to insure a vacuum-tight seal. The gasket 12, which preferably is composed of rubber, is cooled by the water jacket 13. As will be observed, the flange 10 is spaced an appreciable distance from the adjacent internal wall of the retort and thus establishes a passage or section 14, beyond the condenser plate, in which the more volatile metals such as sodium and potassium may condense. Such lighter metals will condense on the wall of the retort close to the cooling jacket 13 in the form of a deposit 15. If desired, a removable sleeve may be inserted in the section 14 and closely adjacent the internal wall of the retort

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upon which such lighter metals may deposit. The sleeve may be removed from the retort, at the end of a cycle to recover or otherwise dispose of the lighter metals condensed thereon. In order to accurately control the condensing temperature a temperature indicating and/or recording element such as the pyrometer 16 is associated with the condenser plate 1 and is adapted to register the temperature in the magnesium condensation zone.

The operation of the reduction unit will have been understood from the foregoing description. In carrying out the process of producing magnesium metal from magnesium ores, calcined dolomite and ferrosilicon are ground to a suitable degree of fineness, mixed in proper proportions and briquetted and the briquettes are charged into the furnace. If a radiation shield is employed it is then inserted in place between the condenser and reducing sections. The retort may be heated in any suitable manner as by gas firing and the charge allowed to burn off for a short period of time sufficient to reduce the portion of combined water in the charge to the desired extent. The retort is then closed by attaching the condenser-closure and bolting the flange 11 to the body of the retort. In ordinary operations the average time under vacuum is about 9½ hours and the retort temperature is of the order of 2150° F. During this period the vapors of magnesium and lighter associated metals such as sodium and potassium are evolved from the charge and pass into the condenser section. During this time the spray 8 is operated and the flow of coolant is adjusted to maintain the condenser temperature, as shown by the pyrometer 16, at between 600° F. to 1000° F. and preferably between the range 800° F. to 1000° F. In these circumstances magnesium metal will deposit in the form of a dense compact mass 17, as shown in the drawings. When a radiation shield is employed it will be understood that the shape of the condensed magnesium muff will be controlled or determined by the design of the shield. During operation the more volatile metals, such as sodium and potassium, do not condense with the magnesium due to the fact that the temperature in the magnesium condensing zone, adjacent the plate 1, is above the volatilization point of these lighter metals. The vapors of such lighter metals therefore flow back into the section 14 and deposit on the cooler section and close to the cooling jacket 13.

At the end of such a cycle the vacuum is broken, the closure plate is removed together with the attached magnesium deposit. The deposit of the pyrophoric metals may be allowed to burn, in situ, or, when deposited on a removable sleeve may be removed with the sleeve and burnt off outside of the furnace or recovered. The residue accumulating in the retort is then removed and the cycle repeated.

It will be appreciated that utilization of the principles of the invention insures an improved operation. By employing a controlled stream or spray of a liquid coolant directed on the condenser plate, rapid and controlled abstraction of heat from the condensing magnesium is insured. As explained, this condensation temperature may be controlled accurately within close ranges to insure the desired crystalline structure of the deposited magnesium as well as a sharp fractionation of magnesium from the lighter metals associated therewith. As has been noted, because of its high latent heat of vaporization, water or

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water solutions constitute the preferred coolant. However, other solutions characterized by a substantial capacity to quickly abstract the heat of condensation may be utilized.

The critical importance of the improved method of condensation will more readily be appreciated from a consideration of the graph shown in Fig. 2. In this graph the amount of magnesium condensed is plotted on ordinates against the time of the distillation cycle on abscissa. It will be observed that the condensation rate is relatively great and at a maximum in the early part of the cycle, i. e., after approximately one hour under vacuum and then diminishes gradually and substantially uniformly to the end of the cycle. In a typical case, therefore, at about an hour after the beginning of the cycle, i. e., at the peak of the curve a maximum abstraction of heat should be insured. Since about 2500 B. t. u. must be abstracted from the magnesium vapors to produce one pound of the metal condensate it will be seen from the graph that the cooling system to be effective, in the depicted case, must abstract a great amount of heat in the relatively short interval of peak load after which the cooling requirements diminish continuously and gradually during the remainder of the cycle. The advantages of positively insuring a regulated or controlled abstraction of heat based on the sharp variation in condensation rate are apparent. The described method under which the volume of coolant utilized per unit of time and hence the degree of cooling may readily and accurately be controlled admirably meets the difficult conditions outlined.

The control of the cooling capacity of the condenser established by adjustment of the volume of coolant from jet 8 may be manual or automatic such, for example, as an automatic time control wherein maximum volume of coolant is supplied at the time or interval of maximum cooling requirements after which at desired time intervals the volume of coolant is correspondingly adjusted to the reduced demands of the condensation cycle.

It is to be understood that while the principles of the invention have been explained with relation to the condensation of a particular metal vapor, i. e., magnesium, the fundamental concepts may be invoked in the condensation of other distillable metals such, for example, as zinc and the like.

It will be understood that other specific designs of condenser units may be employed which are functionally equivalent to that shown, i. e., which invoke the concept of cooling and accurately maintaining the condenser temperature by vaporization of a coolant, particularly one having a high latent heat of vaporization. Therefore, while a preferred modification of the invention has been described it is to be understood that this is given to exemplify the underlying principles involved and to illustrate the advantages therein.

I claim:

1. Apparatus of the class described comprising a retort having a distillation zone and a condensing zone in open communication with the distillation zone, a closure for the retort at the condensing zone, means to maintain the retort under subatmospheric pressure, the closure comprising a removable metallic condenser plate, the inner surface of said closure plate being disposed to be directly contacted by magnesium vapors in the condensing zone, and the outer surface of said closure plate directly opposite to said inner vapor contacting surface thereof, being exposed to the atmosphere, adjustable spray means

positioned in close proximity to said outer surface of said plate and adapted to spray liquid coolant directly on the outer surface of the plate in varying volume to abstract heat therefrom by evaporation at optimum rates to control the temperature of the condensing material and the condensation of magnesium vapors in maximum amount on the said inner surface of the plate in a dense, compact, crystalline mass, said condenser plate being of a diameter substantially less than that of the contiguous section of the retort and disposed inwardly of the outer end of the retort, an annular flange at the periphery of the condenser plate extending outwardly concentrically with said retort and providing a substantial annular space or condensing zone between said annular flange and said retort, outwardly beyond the condenser plate, for condensation of the more volatile metals therein, and a terminal flange parallel to said condenser plate and adapted to engage the outer end of the retort, providing an open dished closure to receive said spray impinged against the outer surface of the condenser plate.

2. Apparatus of the class described comprising a retort having a distillation zone and a condensing zone in open communication with the distillation zone, a closure for the retort at the condensing zone, means to maintain the retort under subatmospheric pressure, the closure comprising a removable metallic condenser plate, the inner surface of said closure plate being disposed to be directly contacted by magnesium vapors in the condensing zone, and the outer surface of said closure plate directly opposite to said inner vapor contacting surface thereof, being exposed to the atmosphere, adjustable spray means positioned in close proximity to said outer surface of said plate and adapted to spray liquid coolant directly on the outer surface of the plate in varying volume to abstract heat therefrom by evaporation at optimum rates to control the temperature of the condensing material and the condensation of magnesium vapors in maximum amount on the said inner surface of the plate in a dense, compact, crystalline mass, said retort comprising a cylindrical portion providing said distillation zone and an adjoining frusto-conical

portion providing said condensing zone and flaring outwardly from said cylindrical distillation zone portion and having a terminal cylindrical portion to engage the wall of the furnace in which the retort is disposed, said removable condenser plate being of a diameter substantially less than that of the terminal cylindrical portion of the retort and disposed inwardly of the outer end of the terminal cylindrical portion of the retort, a cylindrical flange at the periphery of the said plate extending outwardly concentrically with said terminal cylindrical portion of the retort and providing a substantial space or condensing zone therebetween, outwardly beyond the condenser plate, for condensation of the more volatile metals therein, and a terminal flange parallel to said condenser plate and adapted to engage the outer end of the retort, providing an open dished closure to receive said spray impinged against the outer surface of the condenser plate.

3. Apparatus according to claim 2 in which said condenser plate is substantially in alignment with the wall of the furnace, said terminal cylindrical portion of the retort extending outwardly beyond the furnace wall, a cooling water jacket annularly disposed upon the outwardly extending terminal cylindrical portion of the retort at the condensing zone for the more volatile metals, and a gasket disposed between said water jacket and said terminal plate flange.

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