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1,884,993

PRODUCTION OF METALLIC MAGNESIUM

Filed July 14, 1932

Fig 1

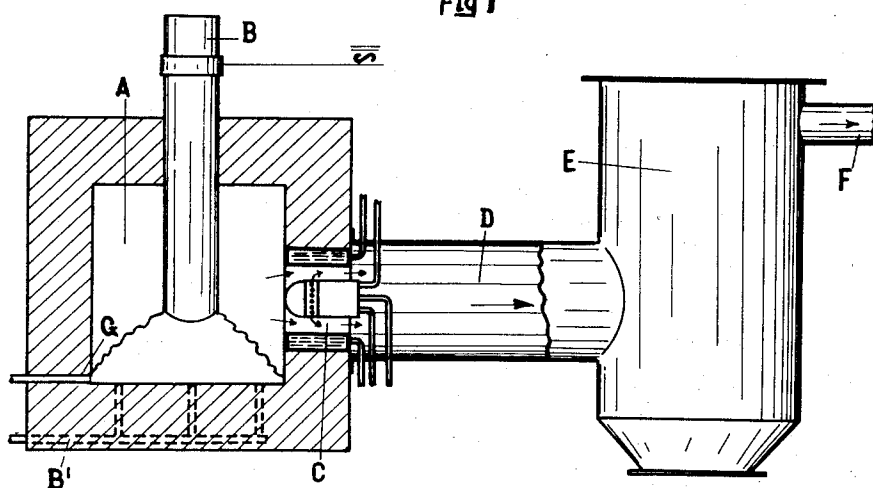
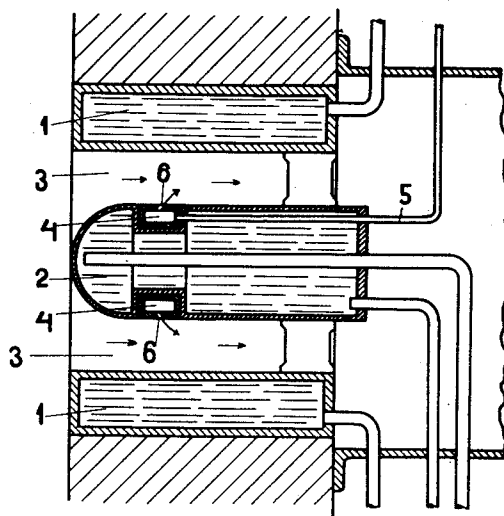


Fig 2



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PRODUCTION OF METALLIC MAGNESIUM

Application filed July 14, 1932, Serial No. 622,553, and in Austria July 8, 1931.

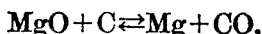
This invention relates to the production of metallic magnesium.

It is an object of the present invention to provide a simple and economical process for producing magnesium by thermal reduction with carbonaceous material of magnesium containing material such as magnesium oxide or raw material containing or yielding magnesium oxide.

Another object is to devise a process for producing magnesium by the thermal reduction method in which the formation of magnesium oxide is greatly minimized.

A further object is to provide a method of producing massive and compact metallic magnesium by the thermal reduction method.

The manufacture of metallic magnesium by way of reducing magnesium containing material with carbonaceous material, to produce, according to the reversible reaction



magnesium vapor besides carbon monoxide, and by condensing the said vapor, presents the difficulty that the metal produced is superficially reoxidized by the reaction gases, more particularly by the carbon monoxide produced during the reaction. The reaction is therefore carried out in an indifferent or reducing atmosphere, preferably in a hydrogen atmosphere, a strong gas current being passed for the purpose of diluting the reaction gases and at the same time quickly removing from the reaction chamber the magnesium vapors generated during the process. Further, it has already been proposed to counteract reoxidation by effecting the condensation of the vapors by sudden chilling.

To obtain an effective dilution of the reaction gases it is necessary to add in the heating zone to the carbon monoxide formed five to ten times the quantity of hydrogen. In addition to the solid reaction mixture, it is necessary therefore to heat to the reaction temperature which, in the case of thermic reduction of magnesium oxide with coal, is between 2000 and 2500° C., also the large quantities of the gas added for the purposes of dilution. This means a not inconsiderable waste of energy. Moreover when the vapors

are condensed to fine magnesium powder by sudden cooling, these highly heated quantities of gas have to be quickly brought down to a low temperature. This again requires an unnecessary consumption of energy.

This invention is based on the fact that at high temperatures the reversible reaction $\text{MgO} + \text{C} \rightleftharpoons \text{Mg} + \text{CO}$ proceeds almost completely in the forward direction (the reaction left to right in the equation), the chemical equilibrium being displaced in the back direction (right to left) only below the reaction temperature. This has led to the assumption that it should be possible, without in any way affecting the successful result, to add the great quantities of gas required for dilution of the carbon monoxide, not as early as in the zone of reaction but only behind the same. Careful experiments have borne out the correctness of that assumption.

Consequently, the process according to the invention consists substantially in maintaining in the reduction chamber such a high temperature that the equilibrium is disposed practically from left to right in the reaction $\text{MgO} + \text{C} \rightleftharpoons \text{Mg} + \text{CO}$, whereby any re-oxidation of the metallic magnesium is avoided with a slight dilution of the furnace atmosphere with hydrogen (or other gas indifferent to, or reducing, magnesium) or even without any addition of such a gas during the reduction, and in diluting the vaporous and gaseous products of the reaction, with considerable quantities of the gases mentioned, only when the said reaction products leave the hot reduction chamber, whereby the metal vapors are cooled down at the same time to the temperature of condensation. This avoids, without in any way affecting the result, the necessity of heating this large gas quantity up to the reaction temperature and of cooling it down again from the said high temperature to the temperature of condensation. The gas current effecting the dilution may advantageously be injected into the vaporous or gaseous mixture of the reaction products at the point where the said mixture is discharged from the hot reduction chamber, in cooled state and in such a quantity

that the dangerous interval of temperature is quickly passed in which there is risk of the magnesium vapors being re-oxidized by the carbon monoxide present.

5 Operating upon these major principles, as stated, it will be understood that a number of modified forms of processes may be employed within the scope of the invention. In one preferred method the magnesium containing material, such as magnesium oxide, is
10 ground and admixed with a suitable reducing agent, such as coke or coal. To this mixture may be added a bonding agent, such as tar or pitch, asphalt and so forth, and the plastic mass then made up in the form of
15 briquettes. These briquettes may then be baked to carbonize the binder. The product thus obtained may be ground and used as a charge for the furnace. If desired, the charge for the furnace may be made up in
20 the form of electrodes containing the ore suitably bonded with the binding agent. However prepared, the mixture of magnesium and the reducing material is then charged to the heating zone of an electric arc and there raised to high temperatures. In
25 carrying out the present process the temperature of the furnace is so regulated that the charge is raised to a high temperature and this temperature is so regulated that preferably
30 the gases and vapors issuing from the furnace are at least at a temperature in the neighbourhood of 2000° C. It is found that in this heating step the hydrogen is not only
35 not necessary but that by eliminating the hydrogen heat economies are obtained which are represented by a saving of 60% in the cost of the electrical energy of the furnace.

40 In the preferred modification of the invention there is introduced into the stream of effluent gases and vapors a stream of cool or cold hydrogen in sufficient quantities to rapidly cool the gases from a temperature of
45 2000° more or less down to a temperature in the neighbourhood of 200° C. Under the cooling action of the indifferent or reducing gas the magnesium is thrown down in solid finely divided form and so-to-speak in stabilized condition. In such a manner during
50 condensation, due to the sudden drop in temperature and the presence of the indifferent atmosphere, reoxidation of the magnesium is substantially eliminated.

55 The gaseous stream from the condenser may be passed through any suitable apparatus for the purpose of separating out any entrained finely divided magnesium powder, and for this purpose filters, centrifugal separators and so forth may be employed.

60 After the preliminary treatment, that is to say after the production of finely divided solid magnesium from the magnesium ore, the product is then treated to reduce it to a massive and compact form. This may be
65 done in a manner described in my copending

application Serial No. 518,915 filed February 27, 1931. In the preferred modification of this second phase of the treatment the finely divided magnesium is vaporized or distilled
70 under reduced pressure and preferably in an atmosphere of an indifferent or reducing gas, such as hydrogen. It will be understood that by distilling magnesium under reduced pressure the vaporization may be effected at lower
75 operating temperatures. Preferably this redistillation is carried out under conditions so controlled as to volatilize and drive off only magnesium vapor and to retain in the distillation vessel or retort such undesired
80 foreign substances as iron, silicon, aluminum, calcium and so forth. Under the conditions of the redistillation under reduced pressure, in the presence of hydrogen, any oxide that may possibly have been formed is removed.

85 The pure magnesium vapor which is evolved in the second distillation stage is then passed to a condensing section and preferably condensed in an atmosphere of hydrogen. This magnesium vapor, in the second
90 condensation step, is in a highly concentrated or dense form as distinguished from the dilute or dispersed condition obtaining in the condenser of the first treatment. Likewise it will be appreciated that the second distillation
95 step operates as a purifying step because by reason of the controlled conditions a so-to-speak fractional distillation of the magnesium may be secured so that pure magnesium uncontaminated with impurities is obtained
100 as a final product.

As pointed out in the prior application, it will be appreciated that the operation may be carried out continuously or the two steps may be aggregated with any desired time
105 interval intervening. Likewise the present process has the advantage, due to the fact that the initial volatilization may be carried out with small batches, of economies of operation which are permitted because of the opportunity to operate the high temperature
110 electrical furnace at the non-peak or low-load periods on the power lines.

115 In the present operation the hydrogen employed in the first or second operation may be withdrawn, cooled, purified, freed from carbon monoxide, and recirculated so as to insure economic operation by reuse of the hydrogen.

120 As in the process described in my copending application referred to, the second compact and purifying step may be carried out by methods other than fractional distillation. For example, the discrete metallic magnesium obtained from the first stage of the
125 treatment may be treated at such a temperature in a reducing or inert atmosphere, such as hydrogen, that the small particles fuse or liquefy and the finely divided magnesium powder flows or coalesces together as a liquid
130

metal. In this form of treatment the temperature is raised above the melting point of the metallic magnesium, that is about 650° C., but below its boiling point. If desired this coalescing step may be performed at reduced pressures but at some point that the temperature at such subatmospheric pressures is not high enough to effect boiling of the coalesced magnesium mass.

While a preferred form of the invention has been described, it will be understood that this is given as illustrative of the underlying principles and not as the exclusive process by which these principles may be effectuated, and the limits of the invention are defined not by the specification but by the scope of the appended claims.

An apparatus which may be used for carrying out the process, is illustrated by way of example in the accompanying drawing.

Figure 1 is a side elevation, partly in section, and

Figure 2 is a detail view on an enlarged scale.

The electric furnace A with the electrodes B, B¹ is connected by a pipe D to a filtering apparatus E. The nozzles 6 for the injection of the diluting and cooling gas (Figure 2) work into the discharge opening of the furnace wall. The gas current is injected in fine jets by means of distributors, the direction of the jets being preferably such as to assist by ejector effect the discharge of the vaporious and gaseous products of reaction from the reduction chamber.

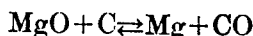
In the construction illustrated, the discharge opening of the furnace is lined with a water-cooled cylindrical jacket 1 into which projects a substantially cylindrical body 2 which is also water-cooled. In an annular groove of the cylinder 2 is mounted a hollow ring 4 to which for instance hydrogen gas is supplied through a pipe 5. The outside wall of the ring is provided with nozzles 6 which are arranged obliquely to the axis of the ring. Close to the bottom of the furnace opens a pipe G (Figure 1) intended for the introduction of a diluting gas into the reaction chamber if desired.

The products of reaction escaping from the furnace become mixed with the cold hydrogen supplied by the nozzles 6 and play on the water-cooled walls of the cylindrical jacket 1 and the body 2, so that a sudden cooling is produced. The fine magnesium powder is separated in the filtering apparatus E, whilst the gas freed from the magnesium escapes through the branch F. After having been freed from the CO formed during the reaction, the gas can be returned to the furnace.

I claim:—

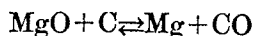
1. A process of producing magnesium which comprises admitting a mixture of magnesium containing material and carbonaceous reducing material to a heating zone to pro-

duce, besides carbon monoxide, magnesium vapors; while keeping the evolved vaporious and gaseous products of reaction out of contact with an extraneous reducing gas and at such a high temperature that the chemical equilibrium of the reversible reaction



is practically disposed from left to right in the equation, and then suddenly diluting and chilling the magnesium vapors at the point of their discharge from the heating zone by direct contact with a cool non-oxidizing gas.

2. A process of producing magnesium which comprises admitting a mixture of magnesium containing material and carbonaceous reducing material to a heating zone to produce, besides carbon monoxide, magnesium vapors; while keeping the evolved vaporious and gaseous products of reaction out of contact with an extraneous reducing gas and at such a high temperature that the chemical equilibrium of the reversible reaction



is practically disposed from left to right in the equation, and then suddenly chilling the magnesium vapors, by mixing a non-oxidizing gas therewith at the point of their discharge from the heating zone, to such a low temperature that thereby any interaction between the metallic magnesium formed by condensation and the carbon monoxide virtually is avoided.

3. A process of producing magnesium which comprises admitting a mixture of magnesium containing material and carbonaceous reducing material to a heating zone to produce, besides carbon monoxide, magnesium vapors, while keeping the evolved gaseous and vaporious products of reaction, in the said zone, out of contact with any substantial amount of an extraneous non-oxidizing gas and at such a high temperature that the chemical equilibrium of the reversible reaction $\text{MgO} + \text{C} \rightleftharpoons \text{Mg} + \text{CO}$ is practically disposed from left to right in the equation and thereby substantial reoxidation of the magnesium vapors is avoided, and injecting into the vaporious and gaseous products of reaction only on leaving the hot reduction zone a materially greater quantity of a cooled non-oxidizing gas sufficient to rapidly transform the magnesium from the vapor to the finely divided solid phase.

4. A process of producing magnesium which comprises heating a mixture of magnesium containing material and carbonaceous material in an electric furnace, while keeping the evolved gaseous and vaporious products of reaction in the furnace out of contact with an extraneous non-oxidizing gas, and at such a high temperature that the chemical equilibrium of the reversible reaction $\text{MgO} + \text{C} \rightleftharpoons \text{Mg} + \text{CO}$ is practically dis-

posed from left to right in the equation, withdrawing the said gaseous and vaporous reaction products from the furnace and condensing the vapors in a separate zone by intimately admixing therewith, at the point of
5 their discharge from the furnace, a cool non-oxidizing gas.

5. A process of producing magnesium which comprises admitting a mixture of magnesium containing material and carbonaceous reducing material to a heating zone to produce, besides carbon monoxide, magnesium vapors, while keeping the evolved gaseous and vaporous products of reaction in the said zone substantially out of contact
15 with an extraneous reducing gas, and at such a high temperature that the chemical equilibrium of the reversible reaction $MgO + C = Mg + CO$ is practically completely displaced from left to right in the equation, withdrawing said reaction products and
20 injecting into them, at the point of their discharge from the heating zone, a non-oxidizing gas in such quantity that the temperature interval is rapidly passed in which there is risk of the magnesium being reoxidized by the carbon monoxide present.

6. A process of producing magnesium which comprises evolving magnesium in vapor form by thermal reduction of a mixture of magnesium containing material and carbonaceous material in an electric furnace, maintaining the heat in said furnace to a degree sufficiently high to retain the evolved
30 vaporous and gaseous products of reaction at a temperature which practically prevents the reoxidation of the magnesium vapors by the carbon monoxide formed during the reduction and without the aid of an extraneous reducing gas, withdrawing the said reaction
40 products from the furnace and injecting into them, at the point of withdrawal, tangentially directed jets of cooled hydrogen in such a quantity that the reoxidation of the magnesium vapor by the carbon monoxide
45 present is substantially avoided.

7. A process of producing magnesium which comprises evolving magnesium vapors by heating, in an electric furnace, a mixture
50 of magnesium containing material and carbonaceous material up to a temperature capable of substantially preventing reoxidation of the magnesium vapors by carbon monoxide formed during the reduction, withdrawing the vaporous and gaseous products from the furnace and injecting into them
55 only at the point of their discharge, a non-oxidizing gas in fine jets which are given such a direction as to assist the discharge by ejector effects and in such a quantity that the
60 magnesium vapors are suddenly condensed to finely divided solid form.

8. An apparatus for producing metallic magnesium which comprises, in combination,
65 an electric furnace adapted to vaporize mag-

nesium from its compound, the furnace being formed with a discharge opening, water cooling means in the discharge opening, a receiving apparatus communicating with the discharge opening; a water cooled ejector
70 mounted within the opening for obliquely ejecting reducing gas into the evolved magnesium vapors for cooling and withdrawing them from the furnace.

In testimony whereof I affix my signature.

FRITZ HANS GIRG.

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