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MANUFACTURE OF METALLIC MAGNESIUM

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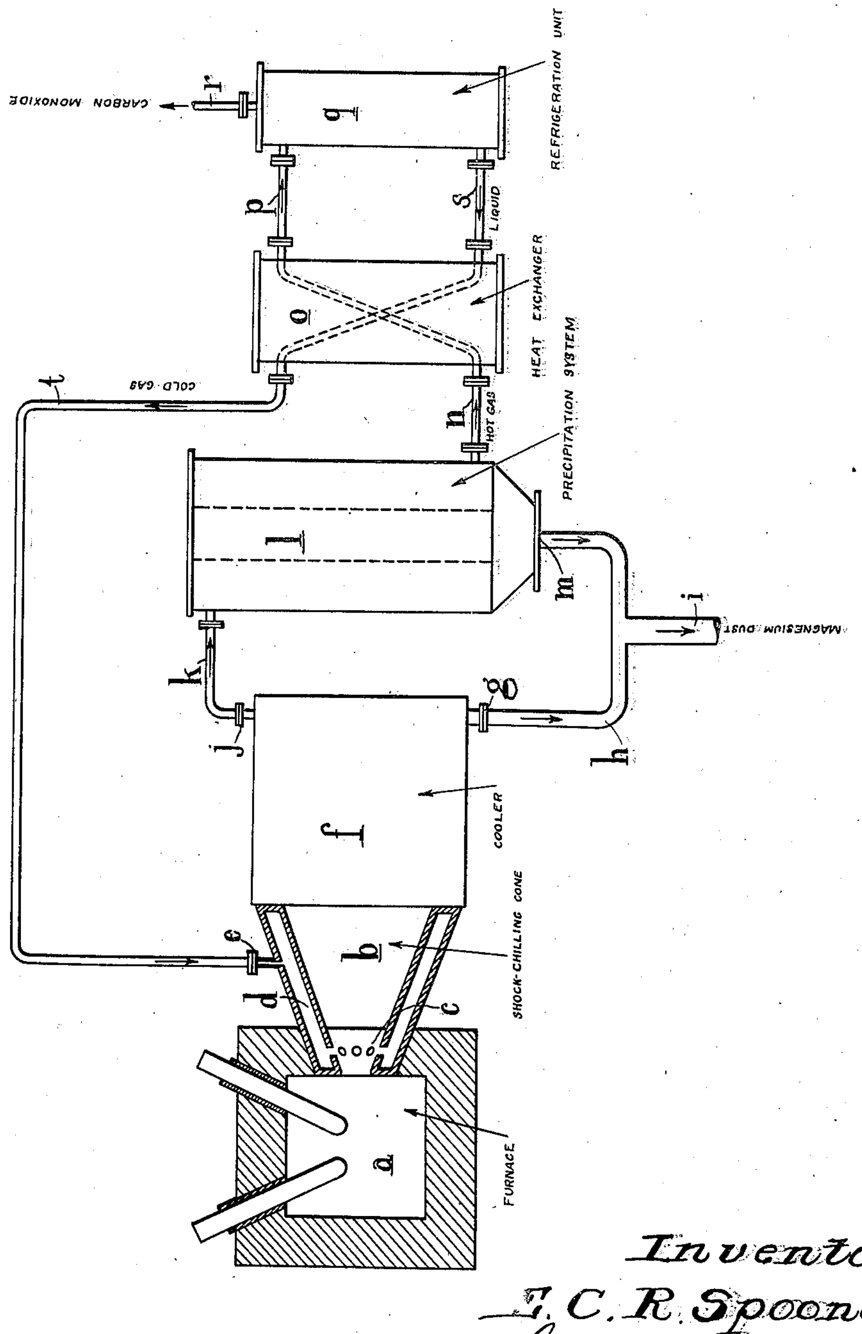

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MANUFACTURE OF METALLIC MAGNESIUM

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6 Claims. (Cl. 75—67)

This invention relates to the manufacture of metallic magnesium from magnesium oxide or from raw materials containing or yielding magnesium oxide by the reversible process according to which magnesium oxide and carbon yield magnesium and carbon monoxide.

The process for the production of the magnesium is carried out at high temperature and in order to prevent the reverse reaction taking place whereby the magnesium would revert to magnesium oxide, sudden chilling of the mixture of magnesium and carbon monoxide must be effected.

According to one known process, this is secured by the addition to the reaction products immediately after leaving the hot reduction chamber of a substantial quantity of hydrogen or any other gas indifferent to or reducing magnesium.

In another process the cooling material now frequently referred to as a shock-chilling medium comprised liquid hydrocarbon oil brought into a state of fine division. The first type of process which may be termed the hydrogen process has its advantages and its disadvantages, and the second type of process which may be termed the liquid oil process has also its advantages and its disadvantages.

An object of the present invention is to provide an improved or modified shock-chilling system retaining largely the advantages of the above two systems and avoiding largely the disadvantages of those systems individually. This can be secured by the use of a suitable condensable gas as the shock-chilling medium.

The invention pertains to a process for manufacturing metallic magnesium by carbothermic reduction of magnesium oxide-containing prime material and shock-chilling of the resulting magnesium vapor-carbon monoxide mixture while using as coolant in a circuit a liquid inert to magnesium, said liquid being vaporized before introduction as a coolant and the vapor egressing being subsequently liquified for the purpose of separating it from carbon monoxide before reintroduction. The improvement on said process according to the invention essentially consists in utilizing the heat content of the outflowing vapor-gas mixture, preferably by contacting it with the returning cooling liquid, for vaporizing the liquid before its reuse as a coolant.

The accompanying diagrammatic drawing illustrates apparatus suitable for carrying the present invention into effect.

In carrying the invention into effect in the form illustrated by way of example there is introduced into the products leaving the furnace or hot reduction chamber *a* in which magnesium oxide and carbon are converted into magnesium and carbon-monoxide a condensable gas having a boiling point within the range +20° to −20° C. and especially butane. The butane or the like enters the shock-chilling apparatus, for instance, a shock-chilling cone *b* in the form of gas. This gas enters the cone *b* through apertures *c*, being led thereto by the shell *d* of the cone and an opening *e*. The products leaving the shock-chilling cone *b* pass to a drum or other cooler *f*, the metal being in the form of dry dust and the shock-chilling medium in the form of gas or vapour. The dust which falls to the bottom of the cooler passes out by way of an opening *g* and pipe *h* to a magnesium dust exit *i*. The mixture of dust and gas or vapour at the top of the cooler *f* passes by way of an opening *j* and pipe *k* to a precipitation system *l* which may comprise dry bag filters or electrostatic precipitators, the exit condition of the dust being dry and the exit condition of the medium being in the form of gas or vapour. The magnesium dust leaves the cooler *l* at the base at *m* and then proceeds to the magnesium dust exit *i* aforesaid. The hot gaseous products from the cooler pass by the pipe *n* to a heat exchanger *o* from which the cold gaseous products leave by way of the pipe *p* and pass to a cooling or refrigeration apparatus *q*. From this apparatus non-liquefied gas leaves at the top by way of the pipe *r* and any liquefied product by way of the pipe *s* which goes back to the heat exchanger *o* from which cold gas ultimately leaves by the pipe *t* leading to the intake *e* to the shock-chilling cone referred to above. Thus as indicated the shock-chilling medium and the carbon-monoxide resulting from the reaction may be separated in a carbon-monoxide removal system of the type effecting condensation by refrigeration and re-evaporation by expansion, the exit condition of the medium being in the form of gas. Where the magnesium dust suspended in the shock-chilling medium is removed by a bag filter system, the temperature of the clean gas leaving the filter system is unlikely to exceed 20–25° C. However, in order to prevent condensation of the medium in the piping and other parts leading to the condensing system, it may be considered desirable to preserve the shock-chilling medium in the gaseous state at all temperatures above normal atmospheric temperature. These considerations in any given instance may indicate desirability of using a shock-chilling medium with a boiling point below 20° C. and preferably below 0° C.

As to the lower limit boiling point permissible, this is dictated mainly by matters of economy. Thus it may be more economic to use a relatively cheap medium of boiling point −20° than an expensive medium of −10° C. In general, however, the increase in condensation costs (refrigeration) as the boiling point falls below °C. is such that the lower boiling point limit may be taken as −20° C.

Among suitable condensable gases, hydrocarbons represent a readily available group, butane as indicated being especially suitable as, for instance, it is a relatively cheap and abundant hydrocarbon, and has a boiling point within the preferred range of 0° C. to −10° C.

The following table contains a number of chosen hydrocarbons:

Hydrocarbon	Boiling point
	°C.
2-methylpropane, C_4H_{10} or $((CH_3)_3CH)$	−10.2
2-methylpropene, C_4H_8 or $(CH_2{:}C(CH_3)CH_3)$	−6
1-butene, C_4H_8 or $(CH_3CH_2CH{:}CH_2)$	−5
1,3-butadiene, C_4H_6 or $(CH_2{:}CHCH{:}CH_2)$	−3
Butane, C_4H_{10} or $(CH_3(CH_2)_2CH_3)$	−0.6 to 0.3
2-butene, C_4H_8 or $(CH_3CH{:}CHCH_3)$	+1 to 2.5
Cyclobutene, C_4H_6 or $(CH{:}CHCH_2CH_2)$	−3 to +1
Methyl-cyclopropane, C_4H_8 or $(CH_3CHCH_2CH_2)$	5
3-buten-1-yne, C_4H_4 or $(CH \vdots CCH{:}CH_2)$	5
1-butyne, C_4H_6 or $(CH \vdots CCH_2CH_3)$	8.6
2,2-dimethyl-propane, C_5H_{12} or $((CH_3)_4C)$	9.5
Butadiyne, C_4H_2 or $(CH \vdots CC \vdots CH)$	10.3
Cyclobutane, C_4H_8 or $(CH_2CH_2CH_2CH_2)$	13
1,2-butadiene, C_4H_6 or $CH_2{:}C{:}CHCH_3$	19

This alternative method of employing a shock-chilling condensable gas simplifies the problem of condensing the medium for the release of the CO, that is to say the cooling or refrigeration unit *q* may take the form of a single gas cooler, for example with bundles of pipes externally water cooled, but at the same time (i) the whole dust precipitation system must be kept hot in order to prevent hydrocarbon condensation in the presence of the dust and (ii) it is necessary to heat and revaporise the medium after condensation before re-using the medium in the gaseous condition for shock-chilling.

It must be understood that these latter disadvantages may outweigh, practically and economically, the advantages of the less costly condensation system in some instances.

General

In the foregoing statements reference has been made, for use as shock-chilling media, only to such hydrocarbons as possess a single boiling point. There is no fundamental objection to the use of mixtures of hydrocarbons but in such cases the boiling point range of the mixture should not exceed 20° C.

The reason for the limitation of the boiling point range, with preference for a medium with a single boiling point, lies in the fact that the difficulties of condensation are in general accentuated where condensation takes place over a wide range of temperature.

Referring to the boiling point limits +20° C. to −20° C. quoted earlier, shock-chilling media within this range include not only those possessing single boiling points but any mixture of such media the boiling point range of which falls within these limits.

I claim:

1. In the process for manufacturing metallic magnesium by carbothermic reduction of magnesium oxide-containing prime material and shock-chilling of the resulting magnesium vapor-carbon monoxide mixture while using as coolant in a circuit a liquid inert to magnesium, said liquid being vaporized before introduction as a coolant and the egressing vapor being then liquified again for the purpose of separating the condensable gas from carbon monoxide before reintroducing it as the coolant; the step of utilizing the heat content of the outflowing vapor-gas mixture for vaporizing the liquid before the reuse as a coolant.

2. In the process for manufacturing metallic magnesium by carbothermic reduction of magnesium oxide-containing prime material and shock-chilling of the resulting magnesium vapor-carbon monoxide mixture while using as coolant in a circuit a liquid inert to magnesium having a boiling point within the range minus 20° C. to plus 20° C., said liquid being vaporized before introduction as a coolant and the egressing vapor being then liquified again for the purpose of separating it from carbon monoxide before reintroducing it as the coolant; the step of utilizing the heat content of the outflowing vapor-gas mixture by contacting it with the returning cooling liquid, for vaporizing the liquid before the reuse as a coolant.

3. In the process for manufacturing metallic magnesium by carbothermic reduction of magnesium oxide-containing prime material and shock-chilling of the resulting magnesium vapor-carbon monoxide mixture while using as coolant in a circuit a liquid inert to magnesium having a boiling point within the range minus 20° C. to plus 20° C., said liquid being vaporized before introduction as a coolant and the egressing vapor being then liquified again for the purpose of separating the condensable gas from carbon monoxide before reintroducing it as the coolant; the step of utilizing the heat content of the outflowing vapor-gas mixture, by contacting it in countercurrent with the returning cooling liquid, for vaporizing the liquid before the reuse as a coolant.

4. A process as claimed in claim 1, in which the condensable gas has a boiling point within the range +20° C. to −20° C.

5. A process as claimed in claim 1 in which the condensable gas is selected from the group of gases consisting of 2-methylpropane; 2-methylpropene; 1-butene; 1,3-butadiene; butane; 2-butene; cyclobutene; methyl-cyclopropane; 3-buten-1-yne; 1-butyne; 2,2-dimethyl-propane; butadiyne; cyclobutane; and 1,2-butadiene.

6. A process as claimed in claim 1 in which the condensable gas is butane.

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