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PRODUCTION OF METALLIC MAGNESIUM

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My invention relates to the production of metallic magnesium.

It is an object of the present invention to provide a novel process to produce compact metallic magnesium.

Another object is to provide an improved and efficient method of preparing magnesium, in which reformation of magnesium oxide is substantially minimized.

Yet a further object is to provide a process for producing metallic magnesium in a purified form.

Other advantages will appear as the description proceeds.

As far back as in the eighties of the last century attempts were made to utilize the method of smelting with carbon, which plays so important a part in zinc technology, for the obtaining of magnesium also. These attempts came to grief, however, owing to the reversibility of the reaction $\text{MgO} + \text{C} = \text{Mg} + \text{CO}$. For a long time it was even regarded as doubtful whether magnesia could be reduced at all with the aid of electrically heated carbon; scientific experiments led finally to the answering of this question in a positive sense (Slade, Journ. Chem. Soc., 1908, pp. 327 et seq.) but no practical achievement followed from this knowledge. On the contrary, in view of the difficulties involved, the technique of magnesium production took a turn, about the year 1915, in the direction of aluminothermic and silicothermic reduction, with the view of avoiding the formation of carbon monoxide from the reducing agent; with the same object in view it was subsequently proposed to employ ferrosilicon and calcium carbide as reducing agents. The problem of smelting magnesium oxide with carbon, the cheapest available reducing agent, which had so long defied solution, was solved in principle only a few years ago, by the adopting of a two-stage process in the first stage of which the mixture of gases and magnesium vapor leaving the hot furnace is abruptly cooled down to a temperature below the solidification point of magnesium, and in this way condensed to powder that is as far as possible free of oxide, and which then admits of conversion in the second stage to liquid magnesium, after elimination of the carbon monoxide present, in different ways but preferably by distilling out the metal in a vacuum.

The invention relates to the conversion of magnesium oxide with carbon in this two-stage process, and consists essentially in heating the magnesium dust obtained in the first stage of the process, after having been separated from

carbon monoxide, in the presence of reducing agents that fulfill the condition of not yielding gases having an oxidizing action upon magnesium, to a temperature at which the reducing agent used withdraws the oxygen from the magnesium oxide still present in its original condition or as re-formed by reversal of the reaction. The magnesium thereby disengaged in the form of vapor is then condensed to a liquid or solid deposit.

By virtue of the fact that what is done in the second stage of the process is not simply to distill out the magnesium present, but to complete the reduction with the aid of reducing agents which yield no carbon monoxide, a better magnesium yield is obtained without rendering the operation appreciably more difficult. The added cost of the more expensive reducing agent of the last-mentioned type that has to be used in the second stage according to the present invention is abundantly outweighed by the improved magnesium yield, in view of the fact that but small quantities of this reducing agent are required in this stage. The additional expenditure in the matter of heat is not considerable, since the reduction temperature for all the reducing agents of this second type is far below the temperature at which the smelting of magnesium oxide with carbon has to be carried out. It is therefore sufficient to heat the mixture of magnesium dust and reducing agent in the second stage of the process to a temperature not very much above the distillation temperature of magnesium at ordinary pressure, it being also possible to work under increased pressure. Suitable reducing agents of this second type are for example aluminum, silicon (or ferrosilicon) and calcium carbide.

Instead of adding the reducing agents of the second type to the magnesium dust only before starting the second stage of the process, it is also possible, with advantage, to generate these reducing agents in the first stage of the process, by adding to the charge, together with a suitable excess of carbon, oxides which react with carbon at the working temperature to form carbides or which are reduced by the carbon to metals capable of reducing magnesium oxide.

According to a preferred embodiment of the invention there are employed as initial material magniferous native products containing suitable oxides as natural concomitants, which is the case more particularly with calcined dolomite, the most readily available magniferous raw material. When starting with this material, at the

temperatures at which the smelting of magnesium oxide with carbon proceeds at a suitable rate (temperatures in excess of 1900° C.), simultaneously with the reduction of the magnesium oxide, there also takes place the formation of calcium carbide from the calcium oxide present, which carbide then acts as a reducing agent in the second stage of the process at temperatures between 1150° and 1500° C. If in lieu of dolomite calcined dolomitic magnesite or calcined magnesite be employed as starting material, the calcium content may be made up to the required amount by the addition of natural or artificial products, or some other oxide of a suitable nature, such as aluminium oxide or silicon dioxide (e. g. in the form of sand), may be added. In all cases it is equally possible to employ mixtures of suitable oxides. Any iron oxide present in the raw material or in the additions thereto has no harmful effect but is useful.

If there be an excess of reducing agent in the mixture at the commencement of the second stage of the process the surplus can be turned to account by the introduction of fresh quantities of magnesium oxide.

The charge consisting of mixtures of the starting material with oxide or oxide mixture, and carbon, may be introduced into the practically empty and highly heated reduction chamber in uniformly small portions in accordance with the method forming the subject of my U. S. A. application Serial No. 755,888. If the starting materials used are rich in magnesium oxide proportionately to the non-volatile concomitants present, it is possible with this method of operation to ensure that the formed reducing agents of the second type are carried along in company with the concomitant substances into the condensate by the vaporous and gaseous reaction products. In practising this method small portions of the charge may for instance be thrown into the empty highly heated reduction chamber in the form of briquettes, or alternatively, the charging mixture may be rendered plastic and slippery by the addition of slight quantities of a liquid binding agent and fed into the reduction chamber from beneath in uniformly small amounts at a time.

If there be employed as reducing agent a metal which has a considerable vapor pressure at the working temperatures required for the smelting of magnesium oxide with carbon, or of which the boiling point lies actually below these practical working temperatures, as is the case for example with aluminium, the metal passes over into the gaseous phase and therefore also into the powdery condensate by direct vaporization during the reduction of the magnesium oxide with carbon. In this mode of carrying out the process the additional heat expenditure for the simultaneous vaporization of the reducing agent in the first stage of the process is counterbalanced by the advantage that the reducing agent can be introduced in the form of impure and coarsely comminuted scrap, for example in the form of aluminium chips, and yet be present in the magnesium dust formed by condensation, in an extremely finely divided state up to that of colloidal dispersion.

I use the term "a non-carbonaceous reducing agent" to exclude the use not only of carbon but also of hydrocarbons.

I claim:

1. A process of producing magnesium which comprises evolving magnesium vapor from oxidic magnesium-containing material by thermal re-

duction with the aid of a carbonaceous matter and suddenly cooling the disengaged vapor so as to condense magnesium in solid powdery form, then separating the powdery condensate from carbon monoxide formed during the reduction and heating said condensate in the presence of a non-carbonaceous reducing agent to a temperature above the boiling point of magnesium enabling the non-carbonaceous reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

2. A process of producing magnesium which comprises evolving magnesium vapor from oxidic magnesium-containing material by thermal reduction with the aid of a carbonaceous matter in the presence of a non-oxidizing gas, suddenly cooling the disengaged vapor so as to condense magnesium in solid powdery form, then separating the powdery condensate from carbon monoxide formed during the reduction and heating said condensate in the presence of a non-carbonaceous reducing agent to a temperature above the boiling point of magnesium enabling the non-carbonaceous reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

3. A process of producing magnesium which comprises extracting magnesium in the vaporous state from oxidic magnesium-containing material by thermal reduction with the aid of a carbonaceous matter in the presence of a non-oxidizing gas, and suddenly cooling the disengaged vapor so as to condense magnesium in solid powdery form, then separating the powdery condensate from carbon monoxide formed during the reduction and admixing said condensate with a non-carbonaceous reducing agent, and then heating the mixture to a temperature above the boiling point of magnesium enabling the non-carbonaceous reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

4. A process of producing magnesium which comprises extracting magnesium in the vaporous state from oxidic magnesium-containing material by thermal reduction with the aid of a carbonaceous matter in the presence of a non-oxidizing gas and suddenly cooling the disengaged vapor so as to condense magnesium in solid powdery form, then separating the powdery condensate from carbon monoxide formed during the reduction and admixing said condensate with a metallic reducing agent, and then heating the mixture to a temperature above the boiling point of magnesium enabling the metallic reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

5. A process of producing magnesium which comprises extracting magnesium in the state of vapor from oxidic magnesium-containing material by thermal reduction with the aid of a carbonaceous matter in the presence of a non-oxidizing gas, while mingling the starting mixture of magnesium-bearing prime material and carbonaceous reducing agent with a volatile non-carbonaceous reducing agent and then shock cooling the escaping gases and vapors to produce,

by condensation, a mixture of finely divided magnesium and reducing agent, then separating the solid condensate from carbon monoxide formed during the reduction and heating said condensate to a temperature above the boiling point of magnesium enabling the metallic reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

6. A process of producing magnesium which comprises extracting magnesium in the state of vapor from oxidic magnesium-containing material by thermal reduction with the aid of a carbonaceous matter in the presence of a non-oxidizing gas, while mingling the starting mixture of magnesium-bearing prime material and carbonaceous reducing agent with aluminium and then shock cooling the escaping gases and vapors to produce, by condensation, a mixture of finely divided magnesium and aluminium, then separating the solid condensate from carbon monoxide formed during the reduction and heating said condensate to a temperature above the boiling point of magnesium enabling the metallic reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

7. A process as defined in claim 5 in which a volatile reducing agent is generated from a compound reducible by carbon during reduction of the oxidic magnesium-containing prime material by the action of carbon.

8. A process of producing magnesium which comprises extracting magnesium in the vaporous state from oxidic magnesium-containing material by thermal reduction with the aid of carbonaceous matter in the presence of a non-oxidizing gas, while adding to the magnesium-bearing prime material, together with an appropriate excess of carbon, substances capable of being converted, at the reduction temperature, by the action of carbon into a non-carbonaceous reducing agent, and regulating the reduction of the magnesium-bearing prime material to be effected by the carbonaceous reducing agent added, in such a manner that the non-carbonaceous reducing agent generated during the reduction is substantially carried along in company with the vaporous and gaseous products evolved by the said reduction, and then suddenly cooling the disengaged vapor so as to produce a mixture of magnesium condensed in a finely divided condition with the non-carbonaceous reducing agent carried over by the vapors and gases evolved; then separating the solid mixture from carbon monoxide formed during the reduction and heating said mixture to a temperature above the boiling point of magnesium enabling the non-carbonaceous reducing agent to withdraw oxygen from the magnesium oxide still present in its original condition or as reformed by reversal of reaction, whereupon the magnesium is recovered by condensation.

9. A process as claimed in claim 8 in which there is employed as initial material a magnesian native product containing a concomitant oxide capable of being converted into carbide at temperatures which are not higher than those at which the smelting of magnesium oxide with carbon proceeds at a suitable rate.

10. A process as claimed in claim 8 in which native calcined dolomite or dolomitic magnesite is used as initial magnesiferous material.

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