A method for producing metallic magnesium by vacuum circulating silicothermic process and apparatus thereof. The method comprises the following steps: heating ferrosilicon to a molten state in a heating container; the molten liquid ferrosilicon and MgO-containing powdered ore are periodically caused to pass through a vacuum container in a circulating manner; MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon; and the Mg vapor obtained is condensed into liquid to be collected.

ferrosilicon is heated to a molten state in a heating container

the molten liquid ferrosilicon and MgO-containing powdered ore are caused to periodically pass through a vacuum container in a circulating manner. MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon.

the Mg vapor obtained is condensed into liquid to be collected.
fluorite → dolomite → ferrosilicon (75% Si) → shatter → calcined → compound → mixed → pressure ball → vacuum thermal reduction → solid state crystalline Mg → purifying → ingotting 

Waste slag → cooled → comprehensive utilization, such as manufacturing construction material

FIG. 1
ferrosilicon is heated to a molten state in a heating container

the molten liquid ferrosilicon and MgO-containing powdered ore are caused to periodically pass through a vacuum container in a circulating manner. MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon.

the Mg vapor obtained is condensed into liquid to be collected

FIG. 2
FIG. 4
METHOD FOR PRODUCING METALLIC MAGNESIUM BY VACUUM CIRCULATING SILICOTHERMIC PROCESS AND APPARATUS THEREOF

FIELD OF INVENTION

[0001] The present invention relates to a method and apparatus for producing magnesium (Mg), and in particular to a method for producing metallic magnesium by vacuum circulating silicothermic process and an apparatus thereof.

DESCRIPTION OF PRIOR ART

[0002] Mg and Mg alloys have such advantages as light in weight, high specific strength, excellent thermal conductivity, easy to recycle, little environmental pollution, or the like. They have great value in applications in the fields like manufacture of transportation facilities (e.g. automobiles), mechatronics, aerospace, national defense, etc. They are known as “the green and environmental protection material of the 21st century”.

[0003] Generally, there are two processes for the industrial production of metal Mg, namely, electrolysis and vacuum thermal reduction. In the vacuum thermal reduction method, ferrosilicon is generally used as a reducing agent, and a horizontal retort is commonly used by means of an external heating periodical reducing process. The vacuum thermal reduction method is also called Pidgeon process.

[0004] Chlorine is generated in the process for producing Mg by electrolysis, and this is accompanied by environmental protection issues which are unable to overcome. Therefore, in recent years, this process gradually gives way to the Pidgeon process.

[0005] The yield of raw Mg in China accounts for about 80% of that around the world, and almost all of the raw Mg is produced by the Pidgeon process.

[0006] The so-called Pidgeon process is a method for producing Mg which is optimized in 1942 by L. M. Pidgeon, a Canadian famous metallurgist, and is named after his name. This process is still in use without fundamental improvement. In this process as shown in FIG. 1, ferrosilicon with Si content of 75% is mixed with MgO-containing calcined dolomite powder in such a manner that they contact with each other in the solid phase. The mixture is put into a horizontal retort which is made from heat resistant steel. The retort is heated externally by flame to promote the chemical reactions among the materials in the horizontal retort. The materials react at a temperature of about 1150~1250 °C, and the vacuum degree is generally lower than 20 Pa. The prior Pidgeon process for producing Mg suffers from the following disadvantages.

[0007] 1. The reactants, i.e., ferrosilicon and calcined dolomite, react chemically in such a manner that they contact with each other in the solid phase, and the reaction rate is slow. In a typical process, the period for the reduction reaction is up to 10~12 hours, and the efficiency is low.

[0008] 2. Since the retort is heated externally by flame, the heat conducts gradually from the outside to inside of the reactor. The period is long, the heat energy is lost significantly, and the utilization of heat energy is low. From the view of point of professional analysis, the utilization of heat energy in a typical process is only about 20%.

[0009] 3. Since the reactor volume is limited by the manner of heating externally, the internal diameter of the horizontal retort is typically not larger than 400 mm, the charge volume for each time is low, and the raw Mg produced in one retort for each time only amounts to 20~30 kg. In addition, it has a large space-occupancy, it is difficult to manage in the field, and it is difficult to achieve large scale production and mechanized production.

[0010] 4. Ferrosilicon with Si element content of 75% is used as the reducing agent. Generally, 1.05~1.20 tons of ferrosilicon is consumed per ton of Mg. That is, 45~100% of Si is wasted, while all Fe element is wasted.

[0011] 5. The reduced Mg vapor condenses directly into solid state crystalline Mg at high vacuum degree. As a result, there is no convenience due to flowing, and it is difficult to collect and release Mg.

[0012] 6. The horizontal retort generally adopts heat resistant steel containing Ni and Cr. The heat resistant steel is expensive, consumes quickly, and results in a high cost.

[0013] 7. There is a serious pollution regarding smoke and dust, which results in a poor working environment and has remarkably adverse effects on the environment.

[0014] 8. It is necessary to manually charge the raw materials, remove the slag, and clean the crystalline Mg, which requires great labor intensity.

[0015] In some granted or published inventions and utility models, modifications have been proposed in view of the above drawbacks of Pidgeon process for producing Mg. The main aspect lies in heating by a clean energy like electric energy, instead of heating by flame. In particular, the heat source comprises heating by resistance plate, heating by resistance materials, inductive heating, or the like. Another modification is to heat internally, instead of heating externally.

[0016] Since the above inventions and utility models do not relate to the solid form of the reactant ferrosilicon, they do not increase essentially the efficiency of reduction reaction for Mg.

[0017] To this end, there is proposed a process for producing Mg by heating internally, such as the technique for producing Mg by heating internally disclosed in the Chinese Patent 95100495.6. In this technique, the calcined dolomite, bauxite, and ferrosilicon with Si content of 75% or more are loaded into an electrical furnace. Under the vacuum condition of 0.01 Pa, the materials are heated via the slag resistance, and MgO is thermally reduced by silicon to refine Mg.

[0018] However, in the Chinese Patent 95100495.6, entitled “A novel process for producing Mg by thermally charging an electrical furnace via silicothermic reduction in vacuum”, the only possibility to involve a liquid reaction lies in that after charging the solid materials, the charging materials will turn to molten state as temperature increases. During the initial stage of reaction, the charging materials still contact with each other in the solid phase. But in the reaction system described in this patent, due to the high vacuum degree, it is impossible to obtain Mg vapor in a form of liquid. Mg vapor may be caused to directly condense into solid state crystalline Mg and thus block the vacuum system. Moreover, it is impossible for this process to realize continuous production.

SUMMARY OF INVENTION

[0019] In view of the drawbacks in the prior art, the present invention discloses a novel method for producing metallic magnesium by vacuum circulating silicothermic process and an apparatus thereof.
[0020] It is an object of the present invention to provide a method and apparatus, which can realize continuous or semicontinuous production of metal Mg, so as to improve the efficiency for producing Mg.

[0021] It is another object of the present invention to shorten the reduction cycle for metal Mg and improve productivity of metal Mg.

[0022] It is yet another object of the present invention to improve the utilization of heat energy.

[0023] It is yet another object of the present invention to no longer use a reduction retort made from heat resistant steel.

[0024] It is yet another object of the present invention to free the limit on the internal diameter of reduction retort and realize a large-sized reactor.

[0025] It is yet another object of the present invention to make full use of two elements of Si and Fe in ferrosilicon, and to realize comprehensive utilization of Si and Fe elements by producing continuously and by producing alloys containing two elements of Si and Fe as by-products.

[0026] It is yet another object of the present invention to condense and collect the obtained Mg vapor in the liquid form, make it easy to control the flow direction of Mg products, and make it convenient to collect and release Mg products.

[0027] It is yet another object of the present invention to reduce smoke and dust pollution, which is favorable to environmental protection.

[0028] The method for producing metallic magnesium by vacuum circulating silicothermic process of the present invention comprises the steps of:

[0029] a first step of heating ferrosilicon to a molten state in a heating container, and maintaining the temperature at 1350–1600°C;

[0030] a second step of passing the molten liquid ferrosilicon and the MgO-containing powdered ore blended therein through a vacuum container periodically in a manner of circulating flow, wherein the vacuum container is separated from the heating container, wherein the vacuum degree of the vacuum container is 350–10000 Pa, and MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon;

[0031] a third step of collecting the liquid Mg, i.e. the Mg vapor released upon condensing.

[0032] In the second step of the method for producing Mg of the present invention, under the action of the vacuum suction and the driving force due to thermal expansion of the filled inert gas, the molten liquid ferrosilicon forms a circulating flow and passes through the vacuum container periodically.

[0033] In the second step of the method for producing Mg of the present invention, the powdered ore is sprayed into the circulating molten liquid ferrosilicon and circulates with the molten liquid ferrosilicon and wherein the vacuum container, MgO in powdered ore chemically reacts with the ferrosilicon liquid and forms Mg vapor which rises to the upper part of the vacuum container.

[0034] In the third step of the method for producing Mg of the present invention, Mg vapor is cooled to a temperature of 650–700°C, and captured by liquid Mg droplets, and thus condenses into liquid Mg to be collected.

[0035] In the method for producing Mg of the present invention, the molten liquid ferrosilicon in said heating container has Si percentage content of 30–65% by weight, wherein the process for producing Mg, solid state or molten ferrosilicon alloy or industrial silicon, which has Si percentage content by weight larger than that of the molten liquid ferrosilicon in the heating container, is regularly added to said heating container to supplement the consumed Si element and improve Si content of the ferrosilicon in the heating container, so that the process for producing Mg proceeds continuously.

[0036] In the method for producing Mg of the present invention, upon the process for producing Mg is complete, one or more of industrial silicon, industrial pure iron, and iron alloy is added to the molten liquid ferrosilicon in said heating container, to adjust the chemical composition of the molten liquid ferrosilicon, thereby producing alloys comprising at least two elements of Si and Fe as by-products during producing Mg.

[0037] In the method for producing Mg of the present invention, in the process for producing Mg, liquid waste slag is discharged regularly from the heating container.

[0038] The present invention further provides an apparatus for producing metallic magnesium by vacuum circulating silicothermic process, which comprises:

[0039] a heating container, which contains molten liquid ferrosilicon;

[0040] a vacuum container, a lower end of which communicates with an upper port of a dipping pipe, wherein a lower port of the dipping pipe is inserted below the liquid level of liquid ferrosilicon contained in said heating container;

[0041] a blowing means, which communicates with said dipping pipe and is capable of blowing inert gas into the blowing pipe;

[0042] In the apparatus for producing Mg of the present invention, said vacuum container is arranged above the heating container, said dipping pipe is placed at the lower side of the vacuum container and communicates with said vacuum container. Said dipping pipe is inserted into said heating container. When said lower port of the dipping pipe is immersed below the liquid level of liquid substance in said heating container, the internal space of said vacuum container and its dipping pipe is isolated from the atmosphere to form an enclosed space. The enclosed space becomes a vacuum vessel under evacuating action. The liquid substance in said heating container is suctioned upward into said dipping pipe and said vacuum container.

[0043] In the apparatus for producing Mg of the present invention, said dipping pipe has at least two manifolds. The blowing nozzle of said blowing means is provided under or aside the first manifold and is capable of blowing the inert gas into the first manifold, so that ferrosilicon liquid rises in the first manifold to said vacuum container, but drops in the second manifold to return to said heating container.

[0044] In the apparatus for producing Mg of the present invention, a condenser is provided on said vacuum container, said condenser communicates with the vacuum container, and said vacuum container is evacuated by a pump system via the condenser, so that Mg vapor is cooled into liquid and falls into a liquid Mg storage means under the condenser.

[0045] In the apparatus for producing Mg of the present invention, said dipping pipe has three manifolds, wherein argon is introduced into one of the manifolds, so that ferrosilicon liquid rises in said one of the manifolds to said vacuum container, but drops through two other manifolds and to return to said heating container.

[0046] In the apparatus for producing Mg of the present invention, a cooling member and a Mg liquid spraying member are provided in said Mg vapor collecting means, for
cooling the collected Mg vapor, and for condensing the collected Mg vapor to form liquid Mg by spraying Mg liquid.  

[0047] In the apparatus for producing Mg of the present invention, at least one plasma heating means, which is capable of heating the substance in the vacuum container, is provided on a sidewall of said vacuum container.  

[0048] In the method for producing metallic magnesium by vacuum circulating silicothermic process and an apparatus thereof of the present invention, it is inventive to separate the reaction chamber (vacuum container) from the reactant storage chamber (heating container), and vacuum environment is only developed in the reaction chamber in which a chemical reaction of reducing MgO by Si occurs. Meanwhile, the reactant storage chamber becomes a container for supplemeting raw materials and discharging waste slag. Liquid or solid state ferrosilicon or industrial silicon is mixed in the reactant storage chamber for supplementing the consumed Si element due to the reducing process. The reactant storage chamber is also used to discharge SiO₂-containing slag which is formed during the reducing process. As a result, the process for producing Mg can proceed continuously, provided that the vacuum is not broken.  

[0049] After the vacuum is formed, an inert gas like argon is blown into the dipping pipe, so that the inert gas expands dramatically at a high temperature. In addition to the suction effect of vacuum on the liquid substance, the liquid reactant rises in the dipping pipe to the vacuum container, and then drops in another dipping pipe by gravity. In this manner, a circulating flow is formed between the vacuum container and the reactant storage chamber, and molten liquid ferrosilicon and the powdered ore which have not subjected to the reaction reaction are continuously suctioned into the vacuum container for chemically reacting.  

[0050] It is well known as a rule in the high temperature metallurgical chemical reaction that the chemical reaction rate between solids is very slow, while a reaction in which a liquid participates in is much faster. Therefore, in the present invention, liquid ferrosilicon is used as a reducing agent, and a liquid phase participates directly in the chemical reaction. If there is a fast and intense flowing mixing for the liquid reagents, the interfacial area of the chemical reaction will increase greatly, thus resulting in a much faster reaction rate than that of a quiet liquid. Therefore, the circulating molten liquid ferrosilicon greatly increases the reaction rate, and improves the production efficiency of the process for producing Mg. As a result, in the present invention, it is possible to use ferrosilicon with a relatively low Si content as the reducing agent, for example by preparing ferrosilicon with Si content of 30–65% by using ferrosilicon with Si content of 75%, ferrosilicon with Si content of 45%, or pure iron. In this way, the applicable range of the reducing agent is enlarged.  

[0051] In the process of reducing MgO by the ferrosilicon alloy, with the consumption of Si element, the content of Si in ferrosilicon gradually decreases, so that the reduction rate also decreases. According to the present invention, by continuously adding a Si-containing substance with high Si content (e.g., high-grade ferrosilicon, industrial silicon, or the like), it is possible to always maintain the chemical reaction rate at a relatively high level to realize continuous production.  

[0052] When the process for producing Mg is complete, a certain amount of ferrosilicon may remain. The composition of remaining ferrosilicon is adjusted as by-products during producing Mg, by adding alloys such as ferrosilicon, industrial pure iron, Si—Al—Fe, industrial silicon, or the like. In this stage, the remaining Si and Fe elements are made full use of.  

[0053] In the reducing process for producing Mg, it is favorable to reduce MgO vapor by improving vacuum degree. However, in case of a too high vacuum degree, Mg vapor is prone to directly condense into powder-like solid state raw Mg, which may block vacuum pipes, damage the related apparatuses, and destroy the continuity of production. In the reducing process of the present invention, a relatively high temperature and a relatively low vacuum degree are applied, which are not only favorable to the fast reduction of MgO vapor, but also favorable to collect Mg vapor in the liquid form. Meanwhile, Mg vapor is captured by spraying liquid Mg, which improves the yield of Mg.  

[0054] According to the method and apparatus for producing Mg of the present invention, the ferrosilicon liquid is internally heating by means of an induction coil and a plasma heating gun, which decreases the heat energy waste and improves utilization efficiency of the energy.  

BRIEF DESCRIPTION OF THE DRAWINGS  

[0055] FIG. 1 is a flowchart showing the Pidgeon process for producing Mg.  

[0056] FIG. 2 is a flow schematic view showing the method for producing metallic magnesium by vacuum circulating silicothermic process of the present invention.  

[0057] FIG. 3 is a structural schematic view showing the apparatus for producing metallic magnesium by vacuum circulating silicothermic process of the present invention.  

[0058] FIG. 4 is a schematic view showing vacuum circulating state of the present invention.  

[0059] The following reference numerals are provided in FIGS. 2, 3, and 4:  

[0060] 101 heating container  

[0061] 102 elevating dip pipe  

[0062] 103 falling dip pipe  

[0063] 104 vacuum container  

[0064] 105 induction coil  

[0065] 106 refractory layer  

[0066] 107 heating container tilting means  

[0067] 108 heating container lifting means  

[0068] 109 molten ferrosilicon  

[0069] 110 argon blow pipe  

[0070] 111 upper feeding chamber  

[0071] 112 lower feeding chamber  

[0072] 113 upper feeding chamber valve  

[0073] 114 lower feeding chamber valve  

[0074] 115 powder delivery pipe  

[0075] 116 plasma heating means  

[0076] 117 slag spout  

[0077] 118 dipping pipe flange  

[0078] 119 vacuum chamber upper sealing flange  

[0079] 201 liquid metal Mg  

[0080] 202 liquid Mg storage retort  

[0081] 203 Mg liquid lifting pipe valve  

[0082] 204 Mg liquid lifting pipe  

[0083] 205 liquid Mg storage retort valve  

[0084] 206 condenser  

[0085] 207 Mg liquid quantitative lift pump  

[0086] 208 dedusting vacuum system connecting pipe  

[0087] 209 vacuum connecting pipe valve  

[0088] 210 Mg liquid spraying nozzle  

[0089] 211 Mg liquid droplet
DESCRIPTION OF PREFERRED EMBODIMENTS

The present invention will be described hereinafter in details in connection with the accompanying drawings to enable the skilled in the art to carry out the present invention by referring to the description.

In the method for producing metallic magnesium by vacuum circulating silicothermic process and an apparatus thereof of the present invention, molten state ferrosilicon is used as the reducing agent, and is caused to periodically pass through a high temperature vacuum container in a circulating manner and reduce MgO-containing powdered ore contained in the molten state ferrosilicon, so as to extract metallic Mg vapor.

As shown in FIG. 2, the method for producing metallic magnesium by vacuum circulating silicothermic process of the present invention comprises the following steps.

In step 501, ferrosilicon is heated to a molten state in a heating container.

In this step, said heating container may be an induction furnace. An induction coil may be provided around the periphery of the induction furnace. Upon being energized, the temperature of ferrosilicon liquid may be maintained at 1350–1600°C in the heating furnace.

In step 502, the molten liquid ferrosilicon and MgO-containing powdered ore are caused to periodically pass through a vacuum container in a circulating manner. The vacuum container is separated from the heating container. MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon.

An elevating dip pipe, as well as a falling dip pipe, is connected at the lower part of the vacuum container, and nozzles of these two dipping pipes are immersed below the liquid level of ferrosilicon in the heating furnace. When the vacuum container is evacuated to 350–10000 Pa, under the vacuum suction action, the ferrosilicon liquid rises to two dipping pipes, and continues to rise to the vacuum container through two dipping pipes. An inert gas (e.g. argon) is introduced into the elevating dip pipe continuously, so that the ferrosilicon liquid in the elevating dip pipe obtains additional lifting force to rise, spew into the vacuum container, and then drop through the falling dip pipe by gravity to return to the heating container. In this way, a circulating flow in which the ferrosilicon liquid runs from the elevating dip pipe to the vacuum container, runs from the vacuum container to the falling dip pipe and back to the heating container, and then rises from the heating container to the elevating dip pipe, is formed.

During the circulating process of the ferrosilicon liquid, the powdered ore is sprayed into the ferrosilicon liquid by using an inert gas as a carrier gas. The powdered ore is mixed with the ferrosilicon liquid, and then trapped by the ferrosilicon liquid in the circulating flow. The place at which the powdered ore is sprayed may be the ferrosilicon liquid in the vacuum container, the ferrosilicon liquid in the heating container, and the ascending/falling dip pipe. When the ferrosilicon liquid and powdered ore stay in the vacuum container, a reduction reaction occurs. As a result, Mg vapor is formed, which escapes to the upper space in the vacuum container and is moved away from the vacuum container under vacuum suction.

Si reduces MgO and then is oxidized to SiO2, which combines with CaO, Al2O3, MgO, etc. in the powdered ore, and continues to circulate with the ferrosilicon liquid. When the circulating speed is relatively low, the liquid slag may float above the ferrosilicon liquid in the heating container. In this case, the slag may be regularly discharged from the heating container manually, mechanically, or under influence of airflow.

The molten liquid ferrosilicon in said heating container is required to have Si content more than 30%. Solid state or molten ferrosilicon with Si content of 75% and industrial silicon may be added to the heating container to supplement the consumed Si element and improve Si content of the ferrosilicon in the heating container, so that the process for producing Mg proceeds continuously.

When the process for producing Mg terminates, ceases, or suspends, the composition of remaining ferrosilicon can be adjusted as required to form the required ferrosilicon products, such as ferrosilicon with Si content of 45%, Si—Al—Fe, or the like, as by-products during producing Mg, and flow out as condensed ingots. Then, molten ferrosilicon with Si content of 3065 is added again to the heating container, in order to carry out Mg production for the next round.

In step 503, the Mg vapor obtained in step 502 is condensed into liquid to be collected.

In this step, the Mg vapor obtained in step 502 is cooled to a temperature of 650–700°C, and is captured by liquid Mg droplets, so that the Mg vapor condenses into liquid Mg to be collected.

In the method for producing metallic magnesium by vacuum circulating silicothermic process of the present invention, the liquid ferrosilicon is used as a reducing agent, so that a liquid phase directly participates in the chemical reaction, and the liquid reaction phases subject to intense circulation, which greatly improves the reaction efficiency. By continuously supplementing a reducing agent with high Si content, the process for producing Mg may proceed continuously or semi-continuously. Moreover, by adding alloy products with relatively high Si content, it is possible to adjust the composition of final remaining ferrosilicon, in order to form the required by-products, such as ferrosilicon with Si content of 45% or Si—Al—Fe alloy.

As shown in FIG. 3, the present invention further provides an apparatus for producing metallic magnesium by vacuum circulating silicothermic process, which comprises:

101 a heating container 101, which contains molten liquid ferrosilicon;
111 a vacuum container 104, wherein an upper port of a dipping pipe extends from a lower end of the vacuum container, and a lower port of the dipping pipe is immersed below the liquid level of liquid ferrosilicon contained in said heating container 101;
112 a blowing means 110, which communicates with said dipping pipe, and is capable of blowing inert gas into the dipping pipe.
113 Said vacuum container 104 is arranged above the heating container 101. Said dipping pipe is placed at the lower side of the vacuum container 104 and communicates with the vacuum container 104. Said dipping pipe is inserted into said heating container 101. Said dipping pipe and said vacuum
container 104 is isolated from the atmosphere to form an enclosed space under the sealing action of ferrosilicon liquid 109 in the heating container 101. The covering of the vacuum container 104 comprises sequentially a steel cover, a liner insulating layer, and a refractory layer. Said insulating layer may be an asbestos plate, paraffin, or Al₂O₃ hollow balls. Said refractory layer may comprise high alumina, corundum, carbon, silicon carbide refractory materials. On the sidewall of the vacuum container 104, it is provided at least one plasma heating means 116 for heating materials in the vacuum container 104 to maintain the reaction temperature. In the present invention, two plasma heating means 116 are symmetrically arranged on the sidewall of the vacuum container 104.

[0114] Said heating container 101 comprises a container with a refractory layer 106 and an induction coil 105 arranged around the periphery of the refractory layer 106. When energized, the induction coil 105 can heat the ferrosilicon 109 with Si content of 30–65% in the heating container 101 to 1350–1600°C, so that the ferrosilicon changes to the molten state. Besides, the induction coil 105 inputs energy to maintain temperature during the process for producing Mg. At the side of the falling dip pipe 103, a slag spout 117 is arranged at the upper part of said heating container 101 for regularly discharging the reacted liquid slag. To facilitate lifting and dumping the heating container 101, the present invention further provides a lifting means 107 and a lifting means 108. Said lifting means 107 is connected with the heating container 101 for adjusting the tilting angle of the heating container to dump the final remaining ferrosilicon liquid. Said lifting means 108 is connected with the heating container 101 for adjusting the height of the heating container 101.

[0115] The molten liquid ferrosilicon in said heating container 101 comes from the outside. As shown in FIG. 3, when started initially, the lifting means 108 lowers the heating container 101. Ferrosilicon liquid is injected into the heating container. Alternatively, solid ferrosilicon is placed into the heating container, and the induction coil 105 is energized to melt solid ferrosilicon. During a continuous process for producing Mg, liquid or solid state ferrosilicon is added near the sidewall of the heating container 101 to supplement the consumed Si element.

[0116] The powdered ore is supplied by a powder delivery pipe 115. The powder delivery pipe can communicate with the heating container 101, the vacuum container 104, and also the elevating dip pipe 102 or the falling dip pipe 103. The powder delivery pipe 115 transfers MgO-containing powder by using an inert gas as the carrier gas. In particular, said powder delivery pipe 115 communicates with an argon supply means and a powder supply means, and the powder supply means is arranged at the front end of the argon supply means.

When argon is introduced, the powdered ore supplied by the powder supply means is sprayed into the heating means 101. As further shown in FIG. 3, said powder supply means comprises at least one feeding chamber, and a valve is arranged between the feeding chamber and the powder delivery pipe 115. In the present invention, two feeding chambers are used, as shown in FIG. 3, namely, an upper feeding chamber 111 and a lower feeding chamber 112. An upper feeding valve 113 is provided between the upper feeding chamber 111 and the lower feeding chamber 112. A lower feeding valve 114 is arranged between the lower feeding chamber and the powder delivery pipe 115. This kind of solid material feeding means can prevent air from entering the vacuum system.

[0117] Said vacuum container 104 is provided above the heating container 101. The vacuum degree of the vacuum container 104 is 350–10000 Pa. Said dipping pipe is located below the vacuum container 104, and is inserted into said heating container 101. The dipping pipe and the vacuum container 104 are sealed by the liquid substance in the heating container to form an enclosed space which is isolated from the atmosphere. Said dipping pipe has at least two manifolds, namely, an elevating dip pipe 102 and a falling dip pipe 103. Alternatively, said dipping pipe has three manifolds, wherein argon is introduced into one of the manifolds, while the molten liquid ferrosilicon 109 in the vacuum container 104 refloows in two other manifolds. As shown in FIG. 3, nozzles of said elevating dip pipe 102 and said falling dip pipe 103 are both below the liquid level of liquid ferrosilicon contained in the heating furnace 101. A blowing nozzle of the blowing means 110 is arranged under or aside said elevating dip pipe 102. The blowing means 110 is capable of blowing an inert gas (e.g. argon) into the elevating dip pipe 102, while the molten liquid ferrosilicon 109 in the vacuum container 104 refloows through said falling dip pipe 103. During production, the lower nozzles of the elevating dip pipe 102 and the falling dip pipe 103 are below the liquid level of the molten liquid ferrosilicon liquid in the heating container 101. Meanwhile, argon is introduced into the elevating dip pipe 102. Under the lifting force due to argon thermal expansion and vacuum suction of the system, the molten ferrosilicon 109 enters the vacuum container 104 through the elevating dip pipe 102, and returns to the heating container 101 through the falling dip pipe 103. FIG. 4 is a schematic view showing said vacuum circulating state. The flow direction of the liquid ferrosilicon is indicated by 303. As shown in FIG. 4, in the vacuum container 104, there is a height difference between the molten ferrosilicon level 301 and the molten ferrosilicon level 302 in the heating container. The molten ferrosilicon level 302 in the heating container 101 is also called a free liquid level. The height difference between 301 and 302 may amount to 2 m or more.

[0118] Both the elevating dip pipe 102 and the falling dip pipe 103 are made up of an upper part and a lower part. The upper part is integrated with the vacuum reactor 104. The lower part is connected with the upper part via a flange 118, to facilitate exchanging the lower part of these two different pipes. An opening is arranged on the top of the vacuum container 104. The opening is connected with a pipe with a sealing flange 119, in order to facilitate opening the sealing flange 119 and preheating or repairing the internal space of the vacuum container.

[0119] When the ferrosilicon liquid 109 stays in the vacuum container 104 at a high temperature vacuum condition, MgO in the powdered ore is reduced to form Mg vapor.

[0120] A Mg vapor collecting means is provided on the vacuum container 104. There may be at least two Mg vapor collecting means. In particular, the Mg vapor collecting means comprises a liquid Mg storage retort 202, a Mg liquid lifting pipe 204 communicating with the liquid Mg storage retort 202, a Mg liquid quantitative lift pump 207 arranged in the pipeline of the Mg liquid lifting pipe 204, a Mg liquid spraying nozzle 210 at the outlet of the Mg liquid lifting pipe 204, and a condenser 206. The condenser 206 communicates with the vacuum container 104. Said vacuum container 104 is evacuated by a pump system via the condenser 206, so that Mg vapor passes through the condenser 206. Specifically, as shown in FIG. 3, the condenser 206 is provided on the top of
said vacuum container 104 and communicates with the vacuum container 104, while said liquid Mg storage retort 202 is arranged under the condenser 206. Said condenser 206 is used to cool Mg vapor generated in the vacuum container 104. The condenser 206 and the liquid Mg storage retort 202 are generally arranged aside the vacuum container 104 and communicate with the vacuum container 104. By arranging the liquid Mg storage retort 202 under the condenser 206, it is ensured that the droplet condensing from Mg vapor just fall into the liquid Mg storage retort 202.

[0121] Said condenser 206 is connected with an evacuating means, so as to evacuate the vacuum container 104 via the condenser 206. As shown in FIG. 3, a dedusting vacuum system connecting pipe 208 is arranged above the condenser 206. A vacuum connecting pipe valve 209 is provided at the place where the dedusting vacuum system connecting pipe 208 communicates with the condenser 206. By opening and closing the vacuum connecting pipe valve 209, it is possible to evacuate the vacuum container 104.

[0122] As shown in FIG. 3, said condenser 206 may use a water cooling system, and is arranged above the liquid Mg storage retort 202. A Mg liquid lifting pipe valve 203 is provided at the place where the Mg liquid lifting pipe 204 communicates with the liquid Mg storage retort 202. By using the above-mentioned structural design, after opening the Mg liquid lifting pipe valve 203 and opening the Mg liquid quantitatively lift pump 207, the liquid Mg 201 in the liquid Mg storage retort 202 can be guided to spray from the Mg liquid spraying nozzle 210. Since the condenser 206 communicates with the vacuum container 104, the condenser 206 is filled with Mg vapor, and the temperature of Mg vapor decreases to 650–700° C. due to absorption of heat of the water cooling means. When Mg liquid droplets 211 sprayed from the Mg liquid spraying nozzle 210 fall into the condenser 206, the Mg vapor contained in the condenser may be captured in the form of droplets and fall into the liquid Mg storage retort 202. The liquid Mg storage retort 202 is provided with a liquid Mg storage retort liquid outlet 213 for releasing the produced liquid Mg.

[0123] A condenser valve 212 may be arranged at the place where said condenser 206 communicates with the vacuum container 104. A valve 205 may be arranged on the top of said liquid Mg storage retort 202.

[0124] By applying the apparatus for producing metallic magnesium by vacuum circulating silicothermic process of the present invention, the process for producing Mg is as follows:

[0125] The temperature is maintained at 1350–1600° C. The molten liquid ferrosilicon with Si content of 30–65% is put in the heating container 101. An induction coil 105 is arranged around the periphery of the heating container 101. Upon being energized, the induction coil 105 heats the ferrosilicon 109 in the furnace to maintain the temperature of ferrosilicon liquid in the heating furnace at 1350–1600° C. The height of the heating container 101 is raised by a lifting means 108. When the lower ports of two dipping pipe 102, 103 are immersed by a certain depth into the liquid level of the molten ferrosilicon 109 in the heating container 101, the vacuum container 104 is isolated from the atmosphere. The vacuum container 104 is evacuated to 350–10000 Pa. Meanwhile, argon is blown into the elevating dip pipe 102. Under the dual action of vacuum suction and lifting force due to argon expansion, the liquid ferrosilicon liquid 109 rises to the vacuum container 104 through the elevating dip pipe 102, and then flows downward through the filling dip pipe 103 to return to the heating container 101. These processes are repeated in this way to form a circulating flow. When the ferrosilicon liquid 109 stays in the vacuum container 104, it reacts with the powdered ore which is also in the vacuum container 104 at a high temperature vacuum condition. Mg vapor is formed, escapes into the upper space of the vacuum container 104, and is drawn to the condenser 206 by vacuum suction. The surrounding condensing means absorbs heat from Mg vapor, and thus Mg vapor decreases in temperature. Under the conditions with a temperature of 650–700° C., Mg vapor is captured by the liquid Mg droplet 211 sprayed from the Mg liquid spraying nozzle 210, turns into liquid Mg, and falls into the underlying liquid Mg storage retort 202. When Si content of the liquid ferrosilicon 109 decreases, some ferrosilicon with Si content of 75% is supplemented, so that the process for producing Mg proceeds continuously. When the process for producing Mg is complete, evacuating is stopped, the liquid Mg 201 (i.e., raw Mg product) in the liquid Mg storage retort 202 is discharged. By adding alloys like ferrosilicon, the remaining liquid ferrosilicon 109 in the heating container 101 is adjusted in composition as required. Then, the lifting means 108 and the lifting means 107 are started to descend and tilt the heating container 101. The remaining ferrosilicon liquid flows out and condenses into ferrosilicon products, as by-products during producing Mg. In the whole process, the liquid slag is discharged from the slag spout 117 regularly.

[0126] In the following, the present invention is illustrated with reference to a specific example. Reference numerals like 601, 602, . . . are used to indicate the sequence.

[0127] 601 An intermediate frequency induction furnace with a power of 8000 kw is prepared as the heating container 101. The internal chamber of the heating container is cone shaped, which has a big top and a small bottom, an internal bottom diameter of 100 cm, and a taper of 0.4 (i.e., in case the height in the vertical direction increases by 1 cm, the internal diameter of hearth increases by 0.4 cm), 2 tons of ferrosilicon with Si content of 75% and 4 tons of ferrosilicon with Si content of 45% are added and melt in the heating container 101, to prepare 6 tons of ferrosilicon liquid with Si content of 55%. The temperature is maintained at 1550° C.

[0128] 602 The inside of the vacuum container 104 and the upper/lower dipping pipes 102, 103 are heated to 1000° C. by natural gas flame. The vacuum container 104 has a diameter of 70 cm and a height of 450 cm. The upper/lower dipping pipes 102, 103 may have an internal diameter of 15 cm and a length of 130 cm, and a silicon carbide refractory material is applied.

[0129] 603 13 tons of MgO-containing powdered ore is prepared. In the powdered ore, the MgO content is 80%, the CaO content is 10%, the Al2O3 content is 10%, and the granularity is 0.012 mm. The powdered ore is stored in the upper feeding chamber 111, and the temperature is maintained at 800° C.

[0130] 604 4000 kg of ferrosilicon with Si content of 75% is melt in another induction furnace.

[0131] 605 The vacuum connecting pipe valve 209 and the liquid Mg storage retort valve 205 are opened, and the Mg liquid lifting pipe valve 203 and the condenser valve 212 are closed. 500 kg of molten metal Mg is fed into the liquid Mg storage retort 202, and the temperature is 700° C. Then, the liquid Mg storage retort valve 205 is closed, and the condenser valve 212 is opened.
When the temperature in the vacuum container reaches 1000°C, the heating flame of natural gas is removed. The two dipping pipes 102, 103 are sealed with an iron sheet at the lower end. The vacuum container 104 and the upper/lower dipping pipes 102, 103 are evacuated by an evacuating pump system through a dedusting vacuum system connecting pipe 208.

When the pressure in the vacuum container 104 reaches 10000 Pa, the heating container 101 is lifted slowly by an heating container lifting means 108, so that the lower ends of the upper/lower dipping pipes 102, 103 are immersed below the ferrosilicon liquid level 302 by 65 cm.

When the sealing iron sheet at the port of the upper/lower dipping pipes 102, 103 melts, the ferrosilicon liquid 109 rises to the upper/lower dipping pipes 102, 103 and the vacuum container 104 under vacuum suction. It is further evacuated so that the pressure in the vacuum container 104 reaches 800 Pa, and this pressure is maintained.

Argon is introduced into the argon blow pipe 110 with a flux of 120 NL/min. Upon entering the elevating dip pipe 102, argon is heated to a high temperature by the ferrosilicon liquid and expands. The ferrosilicon liquid in the elevating dip pipe 102 is driven to rise and enter the vacuum container 104. Then, the ferrosilicon liquid drops through the falling dip pipe 103 and returns to the heating container 101, thus forming a circulating flow.

Cooling water is introduced into the condenser 206 with a flux of 50 kg/s. The temperature of the condenser is maintained at 65°C.

The liquid Mg storage retort valve 205 and the Mg liquid lifting pipe valve 203 are opened, and the Mg liquid quantitative lift pump 207 is started to spray Mg liquid at a speed of 30 kg/min.

The lower feeding chamber valve 114 is closed and the upper feeding chamber valve 113 is opened, so that the powdered ore in the upper feeding chamber 111 enters the lower feeding chamber 112. Then, the upper feeding chamber valve 113 is closed and the lower feeding chamber valve 114 is opened.

The powdered ore is sprayed inside the ferrosilicon liquid 109 in the heating container 101, by using the powder delivery pipe 115 in which argon is used as the carrier gas. The spraying rate is 30 kg/min, and the flux of the carrier gas is 120 NL/min. The spraying continues for 60 min.

The spraying of powdered ore is stopped, and the introduction of carrier gas is also stopped.

The flow rate of argon in the argon blow pipe 110 is increased to 170 NL/min for 10 min.

The flow rate of argon in the argon blow pipe 110 is decreased to 50 NL/min for 10 min.

The slag layer over the ferrosilicon liquid 109 in the heating container 104 is discharge completely from the slag spout 117.

The Mg liquid quantitative lift pump 207 is turned off, and the liquid Mg storage retort valve 205 and the Mg liquid lifting pipe valve 203 are closed.

The liquid Mg storage retort liquid outlet 213 is opened to release a portion of the liquid metal Mg 201 for purifying, alloying, or ingotting.

The liquid Mg storage retort liquid outlet 213 is closed.

621 600 kg of molten ferrosilicon with Si content of 75% is injected into the heating container 101 from another induction furnace.

622 The operations in 612–620 are repeated.

623 600 kg of molten ferrosilicon with Si content of 75% is added again to the vacuum heating furnace 101.

624 The operations in 612–620 are repeated.

625 600 kg of molten ferrosilicon with Si content of 75% is added again to the vacuum heating furnace 101.

626 The operations in 612–620 are repeated.

627 600 kg of molten ferrosilicon with Si content of 75% is added again to the vacuum heating furnace 101.

628 The operations in 612–620 are repeated.

629 600 kg of molten ferrosilicon with Si content of 75% is added again to the vacuum heating furnace 101.

630 The operations in 612–620 are repeated.

631 600 kg of molten ferrosilicon with Si content of 75% is added again to the vacuum heating furnace 101.

632 The operations in 612–620 are repeated.

633 The evacuating is stopped, and the condenser valve 212 and the vacuum connecting pipe valve 209 are closed.

634 Argon is blown from the argon blow pipe 110 for 10 min, and the flux is increased to 150 NL/min.

635 The heating container lifting means 108 is started so that the heating container 101 drops slowly, until the ports of the upper/lower dipping pipes 102, 103 completely separate from the ferrosilicon liquid 109.

636 The liquid Mg storage retort liquid outlet 213 is opened to release all Mg liquid 201 for purifying, alloying, or ingotting.

637 The ferrosilicon liquid 109 in the heating container 101 is sampled to analyze Si content. 320 kg of ferrosilicon with Si content of 75% is added, so that Si content of the ferrosilicon liquid 109 in the heating container 101 is 45%.

638 The powder delivery pipe 115, in which argon is used as the carrier gas, is removed. The heating container lifting means 107 is started so that the heating container 101 turns over to dump the remaining ferrosilicon liquid 109 for ingotting and condensing.

In the actual production, the high temperature slag which is discharged regularly is used to preheat the powdered ore which is to be added to the ferrosilicon liquid by heat exchange, to realize energy recycling. For the convenience of the next operation, a portion of the Mg liquid is kept in the liquid Mg storage retort.

In this embodiment, 5170 kg of Mg is obtained, 5920 kg of ferrosilicon with Si content of 75% is consumed, 4000 kg of ferrosilicon with Si content of 45% is consumed, 6245 kg of ferrosilicon with Si content of 45% is obtained as by-products. That is, for per ton of Mg, 663 kg of Si element is consumed, and 47 kg of Fe element is consumed. In contrast, in the Pidgeon process for producing Mg, for per ton of Mg, 1.2 tons of ferrosilicon is consumed. That is, for per ton of Mg, 900 kg of Si element and 300 kg of Fe element are consumed. In the above embodiments of the present invention, for per ton of Mg, the consumption of Si element decreases by 26%, and the consumption of Fe element decreases by 84%.

After calculation, in the present embodiment, the energy consumption per ton of Mg is 9200 kwh, while the energy consumption per ton of Mg for the Pidgeon process for producing Mg in reducing session is 14400–18000 kwh. Therefore, the energy consumption per ton of Mg decreases by 36–49%.
In the process for producing Mg by reducing of the present invention, the reduction retort which is made from the expensive heat resistant steel is not used, thus leaving out the heat resistant steel material consumption.

In the process for producing Mg by reducing of the present invention, it is possible to realize continuous production by continuously spraying MgO-containing powdered ore, discharging the slag periodically, and adding ferro silicon liquid with high Si content.

The process and apparatus of the present invention is suitable to be large-sized, is easily to be mechanized and automated, reduces the labor intensity, and realizes accurate quantitative operations. Since vacuum circulation is applied in the present invention, liquid ferrosilicon and MgO powder are mixed sufficiently, the reaction interfacial area is greatly increased, the production efficiency is greatly improved, and it represents the development trend of the metallurgical industry.

Although the implementations of the present invention have been disclosed as above, the present invention is not limited to the applications described in the description and embodiments, but can also applied in various fields for which the present invention may be suitable. For the skilled in the art, it is apparent to realize further modifications. Therefore, the present invention is not limited to the specific details and the graphical illustration shown and described herein without departing from the general concept as defined by the claims and their equivalents.

1. A method for producing metallic magnesium by vacuum circulating silico thermic process, characterized in that, the method comprises the steps of:
   a first step of heating ferrosilicon to a molten state in a heating container communicating with the atmosphere, and maintaining the temperature at 1350–1600 °C;
   a second step of passing the molten liquid ferrosilicon and the MgO-containing powdered ore blended therein sequentially through a vacuum container under the dual action of vacuum suction and inert gas driving, and repeating the processes to form a continuous circulating flow, wherein the vacuum container is separated from the heating container, wherein the vacuum degree of the vacuum container is maintained at 350–10000 Pa, and during the molten liquid ferrosilicon and the powdered ore periodically passing through the vacuum container in the continuous circulating flow, MgO in the powdered ore is reduced into Mg vapor by silicon in the molten liquid ferrosilicon;
   a third step of collecting the liquid Mg, i.e. the Mg vapor released upon condensing.

2. The method for producing Mg according to claim 1, characterized in that, in said second step, under the action of the vacuum suction and the driving force due to thermal expansion of the filled inert gas, the molten liquid ferrosilicon forms a circulating flow and passes through the vacuum container periodically.

3. The method for producing Mg according to claim 1, characterized in that, in said second step, the powdered ore is sprayed into the circulating molten liquid ferrosilicon and circulates with the molten liquid ferrosilicon, and wherein in the vacuum container, MgO in the powdered ore chemically reacts with the ferro silicon liquid and forms Mg vapor which rises to the upper part of the vacuum container.

4. The method for producing Mg according to claim 1, characterized in that, in said third step, said Mg vapor is cooled to 650–700 °C. and captured by the sprayed liquid Mg droplets, and thus condenses into liquid Mg to be collected.

5. The method for producing Mg according to claim 1, characterized in that, the molten liquid ferrosilicon in said heating container has Si percentage content of 30–65% by weight, wherein in the process for producing Mg, solid state or molten state ferrosilicon alloy, which has Si percentage content by weight larger than that of the molten liquid ferrosilicon in the heating container, is regularly added to said heating container, or industrial silicon is added directly to said heating container.

6. The method for producing Mg according to claim 1, characterized in that, upon the process for producing Mg is complete, one or more of industrial silicon, industrial pure iron, and iron alloy is added to the molten liquid ferrosilicon in said heating container, to adjust the chemical composition of the molten liquid ferrosilicon, thereby producing alloys comprising at least two elements of Si and Fe as by-products during producing Mg.

7. The method for producing Mg according to claim 1, characterized in that, in the process for producing Mg, liquid waste slag is discharged regularly from the heating container.

8. An apparatus for producing metallic magnesium by vacuum circulating silico thermic process, characterized in that, the apparatus comprises:
   a heating container, which contains molten liquid ferrosilicon comprising Mg powdered ore and communicates with the atmosphere;
   a vacuum container, a lower end of which communicates with an upper port of a dipping pipe, wherein a lower port of the dipping pipe is inserted below the liquid level of liquid ferrosilicon including powdered ore contained in said heating container, wherein the internal space of the vacuum container is sealed by the liquid ferrosilicon including powdered ore contained in said heating container to form an enclosed space, the enclosed space becomes a vacuum environment under evacuating, and the liquid ferrosilicon including powdered ore in said heating container is suctioned upward into said vacuum container through the dipping pipe;
   a blowing means, which communicates with said dipping pipe and is capable of blowing inert gas into the dipping pipe, wherein the liquid ferrosilicon including powdered ore which has been suctioned into said dipping pipe continues to rise to the vacuum container under driving of the thermal expansion of the inert gas, and then drops due to the gravity of the liquid ferrosilicon including powdered ore, so that the liquid ferrosilicon including powdered ore develops a repeated circulating flow between the atmosphere of the heating container and the vacuum environment of the vacuum container.

9. The apparatus for including Mg according to claim 8, characterized in that,
   said vacuum container is arranged above the heating container,
   said dipping pipe is placed at the lower side of the vacuum container and communicates with said vacuum container,
   said dipping pipe is inserted into said heating container, wherein when said lower port of the dipping pipe is immersed below the liquid level of liquid substance in said heating container, the internal space of said vacuum container and its dipping pipe is isolated from the atmosphere to form an enclosed space,
the enclosed space becomes a vacuum vessel under evacuating action, and the liquid substance in said heating container is suctioned upward into said dipping pipe and said vacuum container.

10. The apparatus for producing Mg according to claim 8, characterized in that, said dipping pipe has at least two manifolds, the blowing nozzle of said blowing means is provided under or aside the first manifold and is capable of blowing the inert gas into the first manifold, so that ferrosilicon liquid rises in the first manifold to said vacuum container, but drops in the second manifold to return to said heating container.

11. The apparatus for producing Mg according to claim 8, characterized in that, a condenser is provided on said vacuum container, said condenser communicates with the vacuum container, and said vacuum container is evacuated by a pump system via the condenser, so that Mg vapor is cooled into liquid and falls into a liquid Mg storage means under the condenser.

12. The apparatus for producing Mg according to claim 8, characterized in that, said dipping pipe has three manifolds, wherein argon is introduced into one of the manifolds, so that ferrosilicon liquid rises in said one of the manifolds to said vacuum container, but drops through two other manifolds to return to said heating container.

13. The apparatus for producing Mg according to claim 11, characterized in that, a cooling member and a Mg liquid spraying member are provided in said condenser, for cooling the Mg vapor passing through the condenser, and for condensing the passing Mg vapor to form liquid Mg by spraying Mg liquid, thereby forming liquid Mg to be collected.

14. The apparatus for producing Mg according to claim 8, characterized in that, at least one plasma heating means, which is capable of heating the substance in the vacuum container, is provided on a sidewall of said vacuum container.

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