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PROCESS FOR PREPARATION OF MAGNESIUM FERROSILICON ALLOYS

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ABSTRACT OF THE DISCLOSURE

A process for preparing magnesium ferrosilicon alloys wherein a ferrosilicon and a magnesium silicide are provided, the amount of the magnesium silicide being such to give about the equivalent amount of magnesium desired in the final magnesium ferrosilicon product. The two reactants are heated together at a temperature of from about 1,100 to about 1,450° C. and maintained in the molten state for about 30 seconds to about 25 minutes following which the resultant molten magnesium ferrosilicon product ordinarily is cast into a mold of a predetermined form and solidified.

This invention relates to a novel process for alloy production and more particularly is concerned with a novel process for preparing magnesium ferrosilicon alloys.

Magnesium ferrosilicon alloys are used extensively as a source material for introducing magnesium into molten ferrous melts particularly for use in the nodularization of graphite in cast iron. A popular commercial source material for this use is a magnesium ferrosilicon containing about 9 weight percent magnesium. Normally, this additive material as sold commercially is specified as containing magnesium within the range of from about 7.5 to about 9.5 weight percent magnesium because of the difficulty in maintaning a close control of magnesium content in conventional processes of preparation.

Conventionally in making such magnesium ferrosilicon materials, magnesium ingot is plunged into a ladle of molten ferrosilicon or molten ferrosilicon is poured onto magnesium ingot placed in the bottom of a ladle. Both of these techniques suffer from a number of disadvantages. These include; magnesium recoveries are relatively low, being in the range of only 65 to 75 percent at a maximum; the reaction between magnesium and molten ferrosilicon is somewhat hazardous in that it is violent and accompanied with considerable amounts of pyrotechnics, smoking and fuming; the contained magnesium cannot be held within close limits.

Now, unexpectedly a novel process has been found for preparing magnesium ferrosilicon alloys which overcomes the disadvantages of the present methods employed to prepare these materials.

It is a principal object of the present invention to provide a novel process for preparing magnesium ferrosilicon wherein there is magnesium recovery much higher than realized in processes employed heretofore.

It is also an object of the present invention to provide a novel process for preparing magnesuim ferrosilicon alloys wherein the magnesium content can be maintained within a narrow, readily reproducible range from product to product thereby providing improved quality control to the foundry when such materials are employed in the treatment of ferrous based melts.

It is another object of the present invention to provide a novel process for preparing magnesium ferrosilicon alloys which produces markedly less pyrotechnics, fuming and violet reactivity than presently known processes and thereby is markedly less hazardous than the present techniques.

These and other objects and advantages are realized in accordance with the present novel process wherein magnesium silicide (Mg_2Si) is reacted with a ferrosilicon while the mix reactants are in a molten state.

In the actual practice of the present invention a ferrosilicon and magnesium silicide are provided, the amount of the magnesium silicide being such to provide about the equivalent amount of magnesium desired in the final magnesium ferrosilicon product. The two reactants are heated together at a temperature of from about 1,100° C. to about 1,450° C., preferably from about 1,200° C. to about 1,300° C. and maintained in the molten state for a period of from about 30 seconds to about 25 minutes, ordinarily from about 2 to about 20 minutes and preferably from about 10 to about 15 minutes. This treatment ordinarily is carried out under an inert atmosphere, e.g., substantially anhydrous argon, which assures a minimum or even no undesirable side reaction of the mix reactants. With such an atmosphere ordinarily a minimum pressure equivalent to about the natural atmospheric pressure is used. Preferably, a positive pressure, slightly in excess of atmospheric is used, e.g., from about 15.5 to about 20 pounds per square inch absolute, to assure the maintenance of the inert gas in contact with the reaction mass. Alternatively, an inert pad, covering or other melt protecting means can be used to protect the reaction mixture from contact with air during the heating stage of the operation.

Following the heating period, the resultant molten magnesium ferrosilicon product ordinarily is cast into a mold of a predetermined form and solidified.

Usually in the present novel process, magnesium silicide and a ferrosilicon are placed in a crucible, furnace or other melting apparatus and the assembly then heated to the operating temperature and maintained at this temperature for the disclosed reaction period. If desired, however, magnesium silicide can be added to a molten ferrosilicon charge by introduction onto the surface of or by being rabbled in the melt. Alternatively, molten ferrosilicon can be poured onto solid magnesium silicide positioned in a ladle or other melt retaining apparatus.

Conveniently particulated ferrosilicon and magnesium silicide are used to provide a relatively large surface area per unit mass thereby assuring ready and rapid dissolution upon heating to the molten temperature. Relatively finely divided materials, e.g., minus 100 mesh (U.S. Standard Sieve) are very satisfactory although both fine powders and coarse lumps are equally suitable for use in the present process.

Equipment suitable for use in the process of the present invention is melting, molten metal handling, metal transport and metal casting apparatus employed in foundry and the like metal melting, handling and casting arts.

The present process readily is applicable to the preparation of other magnesium containing agents suitable for use as additives to molten ferrous based melts. These include, for example, nickel, copper, nickel and silicon mixtures and alloys and copper and nickel mixtures and alloys. In the preparation of such materials, magnesium silicide and other specified mix reactants are provided and melted together at appropriate temperatures.

The present novel process readily can be used to prepare the corresponding magnesium containing products having a magnesium content of 3 weight percent or lower and has as high as 35 weight percent or more.

The following examples will serve to further illustrate the present invention but are not meant to limit it thereto.

Example 1.—A charge of about 96 grams of minus 100 mesh (U.S. Standard Sieve) of 50 percent ferrosilicon and about 17 grams of minus 100 mesh magnesium silicide (59.7 weight percent Mg) was loaded into a round cup-shaped graphite crucible and the crucible placed in an induction furnace. The crucible was covered with a graphite lid having a hole at its edge for passage of a thermocouple and a hole in its center having a graphite stirring rod passing therethrough. A Vycor quartz glass beaker was inverted over the crucible, its open top edge resting on top of the furnace. The beaker had a hole in the center of the upwardly extended bottom through which the stirrer shaft extended. A glass tube through the bottom of the beaker extended upward from the outside of the bottom of the beaker. A rubber tube was fitted to this glass tube and to a pressurized argon gas cylinder through appriate pressure and flow controls. This beaker assembly served as a mantle for maintaining an inert gas blanket above the reaction mixture. A thermocouple in a graphite sheath was placed in the crucible through the thermocouple passage in the lid.

A low volume argon flow was introduced and maintained over the charge. A 6-kilowatt high frequency convertor connected to the induction furnace was energized and the crucible and charge heated. The charge was heated to the molten state and raised to a temperature of about 1,250° C. and the convertor then shut off. The resulting melt was stirred for about 30 seconds with the graphite rod and the rod then withdrawn from the crucible. The alloy product mass was solidified and cooled to ambient temperature while maintaining the argon blanket. The cooled ingot was removed from the crucible and found to weight about 112.8 grams. The ingot was analyzed for magnesium and silicon and found to contain 8.5 weight percent Mg and 44.8 weight percent silicon. The theoretical magnesium content as calculated from the reactants charged was 8.98 weight percent. This preparation therefore showed an efficiency of about 94.6 percent based on magnesium recovery.

Example 2.—Using the apparatus and procedure described for Example 1, a charge of about 94 grams of the 50 percent ferrosilicon (minus 100 mesh U.S. Standard Sieve) and about 17 grams of the magnesium silicide (minus 100 mesh) (calculated to provide a magnesium ferrosilicon having about 9.15 weight percent Mg) was heated to about 1,250° C. and maintained at this temperature for about 10 minutes. The cast product resulting from this preparation showed a magnesium content of about 8.1 weight percent magnesium indicative of an 88.5 percent magnesium recovery.

This preparation was repeated with the same size charge and the same reactant concentrations except that a coarser reaction mixture (minus 20 mesh) was used. The resulting product again contained about 8.1 weight percent magnesium or 88.5 percent magnesium recovery.

Another preparation was made using the same coarser reactant materials and following the same procedure except that about 5.4 percent, based on the total charge, of a flux material composed of 34 percent magnesium chloride, 55 percent potassium chloride, 9 percent barium chloride and 2 percent fluorspar was placel on the melt surface. The resulting magnesium ferrosilicon product was substantially the same. The product showed 7.9 weight percent magnesium indicative of a 96.4 percent magnesium recovery.

Example 3.—Using the apparatus and procedure described in Example 1, a number of ferrosilicon and magnesium silicide products were prepared from chunks of the reactant materials about 0.25 inch on a side. The melt was heated to a temperature of about 1,250° C. and maintained at between 1,250 to 1,300° C. for a period of 10 to 12 minutes. Four separate runs were made. Table I summarizes the results of these preparations.

TABLE I

Run No.	Magnesium in Charge, Wt. percent	Magnesium in Ingot, Wt. percent	Magnesium Recovery, Percent
1	8.93	7.5	84.0
2	8.93	7.8	87.4
3	8.93	7.3	81.8
4	9.15	7.7	84.3

The products from runs 1, 2 and 3 were used in the treatment of separate melts of cast iron at 2,750° F. following conventional plunging techniques. The results of these tests are summarized in Table II which follows.

TABLE II

Run No.	MgFeSi (grams)	Magnesium Equivalent (grams)	Cast Iron (grams)	Dose Magnesium, percent,	Magnesium Residual in Iron, percent	Magnesium Recovery in Cast Iron, percent
1a	21.35	1.60	1,529	0.105	0.030	28.7
2a	21.37	1.67	1,520	0.110	0.030	27.3
3a	21.38	1.56	1,566	0.100	0.027	27.1

These studies illustrate the exceptionally close quality control and residual magnesium contents resulting when molten cast iron is treated with magnesium ferrosilicon alloys prepared in accordance with the process of the present invention.

Example 4.—A reactant mixture of about 28.35 grams of magnesium silicide (Mg_2Si containing about 59.7% Mg), about 25.05 grams of 50 percent ferrosilicon and about 46.60 grams of 85 percent ferrosilicon, all of minus 20 mesh, was melted in the apparatus described in Example 1 by heating to a temperature of about 1,250° C. and maintaining this temperature for about 10 minutes. The magnesium in the charge was calculated to be 16.92 weight percent. Analysis of the resulting solidified, cooled ingot showed 16.1 weight percent magnesium, i.e., a 95.2 percent magnesium recovery.

This run was repeated except the reaction charge was changed to contain about 47.25 grams of the Mg_2Si, about 18.45 grams of the 50 percent ferrosilicon and about 34.50 grams of the 85 percent ferrosilicon and the actual melting of the charge was carried out at a temperature of about 1,200° C. for a period of about 10 minutes. This run was designed to give a high magnesium content alloy (calculated to be 28.2 weight percent magnesium) particularly suitable for deep plunging treatment of ductile iron.

The ingot analyzed 26.7 weight percent magnesium which is a 94.7 percent magnesium recovery.

It is to be noted that in both of these runs, although products with high magnesium contents were produced there was substantially no violence, excessive fuming, pyrotechnics or metal splattering during the melting operation. The process was smooth and proceeded in a controlled, active manner.

Example 5.—The same apparatus as described in the preceding examples was employed in the preparation of magnesium ferrosilicon. The manipulative procedure was changed in that the minus 20 mesh particulate 50 percent ferrosilicon was charged to the furnace, melted and brought to a temperature of about 1,300° C. under an argon atmosphere. Magnesium silicide (minus 20 mesh) was added, stirred into the melt and the resultant mix held at temperature for about three minutes. The charge was then cooled and solidified into a billet of magnesium ferro-

silicon. The results of two separate runs are summarized in Table III which follows:

TABLE III

Run No.	Furnace Charge Ferro-silicon (g.)	Magnesium Silicide (g.) percent	Magnesium in Charge, weight percent	Magnesium in Product, weight percent	Magnesium Recovery, percent
1	94	14.41	7.94	6.3	79.4
2	94	15.60	8.51	6.8	79.9

This example illustrates the flexibility of process mechanics that can be practiced with the present invention.

In a manner similar to that described for the foregoing examples the present process can be used to prepare magnesium-nickel-silicon alloys, magnesium-copper-silicon alloys and magnesium-copper-nickel-silicon alloys having magnesium contents within a narrow, predetermined concentration range.

CONTROL EXAMPLE

The same apparatus as described in Example 1 was used to prepare a magnesium ferrosilicon alloy in accordance with conventional processes. About 9.5 grams of magnesium granules (minus 14 to plus 20 mesh U.S. Standard Sieve size range) was placed in the bottom of the furnace and about 90.5 grams of 50 percent ferrosilicon (minus 20 mesh) placed on top of the magnesium. This charge was heated to 1,300° C. and held at this temperature for about 10 minutes and then cooled and solidified. During this time the charge was being heated at 1,300° C., there was evidence of considerable fuming, i.e., white smoke, and metal sparking. The ingot product resulting from this process had a number of large hollow spaces on the bottom. Analysis of the ingot indicated a magnesium concentration of 5.7 weight percent. This represented a magnesium recovery of only 60 percent.

Various modifications can be made in the process of the present invention without departing from the spirit or scope thereof for it is understood that we limit ourselves only as defined in the appended claims.

We claim:

1. In a process for preparing magnesium ferrosilicon alloys by reacting a magnesium metal source with a ferrosilicon the improvement which comprises;
   (a) providing a reaction mixture of a ferrosilicon and magnesium silicide, said magnesium silicide being present in an amount to provide an amount of magnesium ranging from about 3 to about 30 weight percent in the resulting ferrosilicon product,
   (b) heating said mixture at a temperature of from about 1,200 to about 1,300° C. for a period of from about 30 seconds to about 25 minutes,
   (c) casting the resulting molten magnesium ferrosilicon product, and
   (d) recovering said magnesium ferrosilicon product.

2. The process as defined in claim 1 and including the step of maintaining the reaction mixture under an inert gas atmosphere during the heating period of process operation.

3. The process as defined in claim 2 wherein substantially anhydrous argon at a positive pressure, slightly in excess of atmospheric is used as the inert gas.

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