

**[54] POROUS REFRACTORY BODY
IMPREGNATED WITH MAGNESIUM****[75] Inventors: Gerald Kotler, Hightstown; Jairaj
Easwaran, Cranbury, both of N.J.****[73] Assignee: NL Industries, Inc., New York, N.Y.****[22] Filed: July 13, 1972****[21] Appl. No.: 271,364****[52] U.S. Cl. 75/58, 75/130 A****[51] Int. Cl. C21c 7/00****[58] Field of Search 75/53-58, 130 A****[56] References Cited
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Primary Examiner—L. Dewayne Rutledge
Assistant Examiner—Peter D. Rosenberg**[57] ABSTRACT**

A composition of matter useful for treating a ferrous melt to reduce sulphur content thereof comprising a compressed porous refractory body of an alkaline earth metal oxide containing a ceramic binder, said body impregnated with magnesium, the particle size of the alkaline earth metal oxide being less than 4 mesh, said refractory body containing at least about 2 parts by weight of alkaline earth metal oxide for each part of binder and said body being impregnated with at least 35 percent by weight of magnesium, based on the total weight of the impregnated composition.

This composition of matter is produced by admixing -4 mesh particles of alkaline earth metal carbonate with a binder, forming pellets of this mixture and firing the pellets to volatilize the carbon dioxide formed. The fired pellets are then immersed into molten magnesium to impregnate the pellets with magnesium metal.

18 Claims, No Drawings

POROUS REFRACTORY BODY IMPREGNATED WITH MAGNESIUM

BACKGROUND OF THE INVENTION

In the iron and steel industry, it is necessary to treat the ferrous base metals while in the molten state with a desulfurizing agent to reduce the sulphur content of the metal product. Magnesium metal is a powerful deoxidizer and desulfurizer. However, magnesium metal boils at a low temperature and therefore the sudden increase in volume which is produced when metallic magnesium is added to the molten iron may result in violent explosions as the magnesium metal is vaporized.

Various methods have been used to reduce this violent activity by slowly introducing the magnesium metal into molten ferrous metal under rigidly controlled systems. Other methods for reducing the violence is to impregnate porous bodies with magnesium metal and to introduce these magnesium impregnated porous bodies into the molten ferrous metal. Under these conditions, the impregnated magnesium metal is released at a slow enough rate that the violence is held to a minimum.

Among the known porous bodies which have been used with limited success for this purpose are porous coke, carbon, graphite, sponge iron and ceramic bodies such as quicklime, lump limestone or dolomite and the like.

It has been found that the porous compositions of the instant invention possess advantages which are not present in the prior art porous bodies.

SUMMARY OF THE INVENTION

A new composition of matter has been prepared comprising a porous refractory body of an alkaline earth metal oxide containing a ceramic binder, said body impregnated with magnesium, the particle size of the alkaline earth metal oxide being less than 4 mesh, said refractory body containing at least about 2 parts by weight of alkaline earth oxide for each part of binder, and said body impregnated with at least 35 percent by weight of magnesium based on the total weight of the impregnated body. Such a product is useful for desulfurizing ferrous melts.

Products produced by the instant invention generally contain from about 30 percent to about 50 percent alkaline earth metal oxide, from about 1 percent to 15 percent binder and from about 35 percent to about 70 percent magnesium impregnated into the pores of the product, these percentages are based on the total weight of the impregnated body.

The porous refractory body contemplated in the instant invention is produced by admixing a particulate alkaline earth metal carbonate and a ceramic binder, pelletizing the mixture, firing the pellets to convert the metal carbonate to metal oxide and to volatilize the carbon dioxide formed and then immersing the fired pellet in molten magnesium to impregnate the pores of the refractory body with magnesium. The amount of alkaline earth metal carbonate employed is from about 75 percent to about 99 percent while the amount of binder employed is from about 1 percent to about 25 percent, all of the percentages are based on the weight of the mixture.

It is intended that the instant invention also contemplates preparing compositions which may fall somewhat outside the lower and upper limits specified above and therefore, these specified limits are merely the preferred range of compositions and should not be construed as being the overall limits contemplated by the instant invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The porous compositions of the instant invention which are infiltrated with magnesium are superior to the porous bodies of the prior art. The instant porous body not only may take up and retain magnesium in amounts greater than about 35 percent of its total weight, but in addition, contains alkaline earth metal oxides which are useful because of their fluxing properties.

In addition to producing a product which has all of these advantages, the porous bodies may be made with inexpensive raw material in an economical manner.

In preparing the porous composition of the instant invention inexpensive granulated alkaline earth metal carbonates, such as limestone, dolomite and similar raw materials may be used. One very inexpensive raw material which may be used successfully is oolitic sands which are found in nature in large quantities. Oolitic sands in general contain from 70 percent to 95 percent calcium carbonate with the remainder usually being alumina and silica. It is necessary to use a particulate alkaline earth metal carbonate, not a massive body. If the source of the alkaline earth metal carbonate is in a massive form, it must be crushed into a particulate form before using.

The alkaline earth metal carbonate raw material is particulate material (or as a massive body which is pulverized) which has an average size range below 4 mesh. Such particulate material is thoroughly mixed with a ceramic binder in amount from about 1 percent to about 25 percent by weight. Most any well-known ceramic binder may be used including clays, e.g., bentonite, water glass, Portland cement and the like. These binders may be used singly or in combination with one another.

When some sands are employed, the sands themselves may contain sufficient amounts of clay which will act as the binder. In such cases it may not be necessary to add the binder as a separate ingredient. In any event however, the presence of the binder is necessary to form a cohesive compressed product.

The alkaline earth metal carbonate and the binder are then mixed with sufficient water (about 2 percent to 6 percent) to form a moldable clay-like texture. This moist mixture is then pressed into pellets or briquettes at pressures from about 1,500 psi to about 30,000 psi. The pressed bodies are then dried preferably at 100° to 250° C. Periods of 2 to 24 hours have been found to be sufficient to remove the moisture.

The dried pressed bodies are then calcined at a temperature from about 875° C to about 1,450° C for 1 to 10 hours. The use of temperatures which lie somewhat below or above this specified temperature range is also contemplated in the instant invention. The temperature range specified above merely is the preferred temperature which may be employed. Two different types of products are obtained when low or high temperature ranges are employed. Using a lower temperature range

of about 875° C to about 1,100° C produces a product which decomposes relatively rapidly during the desulfurization treatment. The magnesium is released rapidly, thus increasing the rate of desulfurization. In contrast, using a high temperature range of about 1,100° C to about 1,450° C produces a relatively more sintered product which is more mechanically stable while the magnesium is being released. With these two types of products it is possible to control the rate of desulfurization within wide variations.

When the low temperature product is prepared, it is necessary to plunge the product into molten magnesium while still hot, i.e., about 750° to 850° C. Periods of from about 2 to about 15 minutes have been found to be adequate to complete the infiltration. The low temperature product should not be cooled below 700° C before introduced into the molten metal.

With either product, however, from about 35 percent to about 70 percent by weight of magnesium is absorbed into the pores of the calcined body, the percentage of magnesium absorbed is based on the total weight of the infiltrated refractory body. The molten magnesium metal penetrates into the pores of the calcined body and remains there upon cooling.

The magnesium infiltrated refractory body when removed from the molten magnesium must be protected from an oxidizing atmosphere during cooling. One particularly satisfactory method is to plunge the hot refractory body in oil during cooling. The cooled refractory body also should be protected from the atmosphere and moisture during storage and shipping.

The ceramic porous bodies prepared in the instant invention may be impregnated with either substantially pure magnesium or magnesium alloys. Alloys particularly desirable to use are magnesium alloys containing calcium, sodium, lithium and mixtures of these metals. The term "magnesium" hereinafter referred to is meant to include magnesium metal and alloys of magnesium metal.

In addition to preparing pellets by placing the mixture into a mold and subjecting the molded product to high pressure, the mixture may be subjected to extrusion and then cut into pellets.

In this extrusion method, the alkaline earth metal carbonate of -4 mesh in size is mixed preferably with from about 10 percent to about 25 percent clay and to this mixture is added from about 2 percent to about 6 percent water. The mixture is then extruded through a die.

The extruded material is then cut into pellets and fired in the manner described previously.

The novel feature of the magnesium infiltrated porous structure produced in the instant invention possesses the following combined advantages over the prior art:

1. have high porosity and therefore are capable of retaining large quantities of magnesium metal;
2. have the composition which combines the features of having alkaline earth metal oxide present for fluxing properties combined with the presence of magnesium which is useful for desulfurizing molten iron metal;
3. the impregnated pellets produced may be structurally strong and capable of withstanding high temperatures. These pellets are those fired at high temperatures;
4. or the impregnated pellets produced may be very active when introduced into molten iron, thus capable

of desulfurizing rapidly the molten iron. These pellets are those fired at the lower temperatures;

5. the magnesium infiltrated pellets made by the instant invention are uniform in composition and when they are used to desulfurize molten iron reproducible results are obtained.

It has also been discovered that when -4 mesh alkaline earth metal carbonate is admixed with a binder and the mixture is briquetted and calcined, the carbonate is decomposed to oxide and the carbon dioxide formed is removed leaving a porous structure of the oxide and the binder. This porous structure remains in substantially the same size and shape as the briquetted mixture before calcination. Apparently the presence of the binder holds the structure together as the carbonate is decomposed to oxide. This porous structure is therefore capable of holding large quantities of magnesium metal or alloy in its interstices.

In order to describe more fully the instant invention, the following examples are presented:

EXAMPLE 1

In this example, a calcium carbonate sand briquette was prepared. The briquette contained 5.0 percent by weight of bentonite clay.

1,682 grams of calcium carbonate sand were mixed with 84 grams of bentonite clay. The calcium carbonate sand contained 90 percent calcium carbonate by weight, the remainder was silica and alumina. The average particle size of the sand was -60 mesh while the bentonite clay was -325 mesh.

15 grams of water were added to agglomerate the mixture. The wetted mixture was placed in a cylindrical mold 1¼ inches in diameter and 4 inches deep. A hydraulically actuated plunger was used to compress the mixture at 7,500 psi for one minute. A cylindrical briquette having dimensions of 1¼ inches in diameter and 2 inches in height was formed. The briquette was removed from the mold and dried in an oven for 24 hours to remove the water. The briquette was then fired at 1,100° C in a kiln for 3½ hours to release CO₂ thereby converting the calcium carbonate to CaO. The briquette however, was strong enough to retain its size and shape. After calcination, the briquette weighed 49.8 gms. The average particle size of the calcium oxide formed in the compressed product was -60 mesh.

In order to impregnate the briquette with magnesium metal, the briquette was removed from the kiln and while still hot was immersed in a bath of molten magnesium at 800° C for about 1 minute. The briquette was quickly drawn below the surface of the molten magnesium and the level of magnesium dropped sharply indicating infiltration of the briquette. After the briquette was removed from the molten magnesium, it was immediately quenched in a bath of oil to prevent oxidation. The cooled impregnated briquette was then placed in a sealed container for storage. Upon analysis of a sample of the briquette, it was found that 45.0 gms. of magnesium had been infiltrated. This amount is equal to 47 percent magnesium by weight of the infiltrated briquette.

In order to illustrate the use of these magnesium infiltrated compositions, the briquettes or pellets prepared according to the procedure described in Example 1 were produced and were added to molten iron in order to desulfurize the iron as follows:

397 lbs. of iron were melted in a furnace at 1,480° C and contained the following analysis:

Carbon - 3.52 percent, Si - 1.72 percent, S - 0.047 percent, P - 0.029 percent. A treating ladle was preheated to 1,480° C and the hot metal was tapped into the ladle. A plunging bell assembly was preheated and 495 gms. of the magnesium infiltrated pellets described above were placed in the bell. A steel plate was placed under the pellets and was secured to the walls of the bell with steel wires. The plunging temperature of the molten iron was allowed to drop to 1,400° C and the bell was plunged at high speed into the iron. After 2½ minutes, the plunging reaction was over and the bell was raised. Spectrographic buttons were cast and analyzed for sulfur. The sulfur content was found to be .10 percent. The retained magnesium in the iron was 0.020 percent.

The desulfurization efficiency was 81 percent while the efficiency of magnesium utilization was 22 percent.

It has been found that the time of immersion of the porous refractory body in the molten iron preferably should be from 1 to 10 minutes to desulfurize the molten iron.

EXAMPLE 2

In this example another calcium carbonate sand briquette was prepared. The briquette contained 2.35 percent by weight of bentonite clay.

1,682 grams of calcium carbonate sand were mixed with 38 grams of bentonite clay. The calcium carbonate sand and the bentonite clay were the same as those described in the previous example.

15 grams of water were added to agglomerate the mixture and pellets were made in the same manner as those described above except that a pressure of 1500 psi was used instead of 7,500 psi. The pellets were fired at 900° C in a kiln for 3½ hours to release CO₂ thereby converting calcium carbonate to CaO. The briquette, however, was strong enough to retain its size and shape. After calcination the briquette weighed 40 gms.

The briquette was impregnated with magnesium metal in the same manner as that described in Example 1. The impregnated briquette was weighed and it was found that 45 gms of magnesium had been impregnated. This amount is equal to 53 percent magnesium by weight of the infiltrated briquette.

When the briquette was used to desulfurize molten iron, the infiltrated magnesium was released more rapidly than that obtained in Example 1. The extent of desulfurization however, was substantially the same.

EXAMPLE 3

The procedure of Example 2 was followed except that a pressure of 7,500 psi was used to compress the sand mixture instead of 1,500 psi.

After weighing, it was found that 52.4 gms of magnesium (54 percent) had been infiltrated into a briquette that weighed 45.2 gms after calcination.

In addition to the process described above in which a particulate alkaline earth metal carbonate material is mixed with a ceramic binder, it has been found desirable to add in addition to the alkaline earth metal carbonate and the binder, a finely divided cellulosic material such as sawdust, corn cob grits, corn stalks, oat hulls and the like which have been ground to -20 mesh.

The addition of this cellulosic material produces a product which is more porous than the previous product and therefore the product is able to absorb more molten magnesium.

When the cellulosic material is employed, the alkaline earth metal carbonate should be present in the mixture in amount from 55 percent to 75 percent, the binder present in amount from 1 percent to 15 percent and the cellulosic material in amount from 10 percent to 30 percent.

The following example illustrates the preparation of calcium oxide pellets which are prepared by admixing calcium carbonate, a binder and a cellulosic material and calcining the mixture to form the product.

EXAMPLE 4

This example illustrates the preparation of a calcium carbonate sand briquette which contains 2.4 percent by weight of bentonite clay and 11 percent by weight of sawdust.

788 gms of calcium carbonate sand as used in Example 2 was hand mixed with 38 gms of 325 mesh bentonite clay and 110 gms of 100 mesh sawdust. The procedure of Example 1 was followed to produce the briquette.

After weighing the briquette, it was found that 27.9 gms of magnesium (66 percent) metal had been infiltrated into the briquette. The infiltrated briquette weighed 14.6 gms after calcination.

EXAMPLE 5

This example illustrates an extrusion process for the production of magnesium infiltratable pellets comprising 85 percent calcium carbonate sand, 10 percent Tennessee ball clay and 5 percent bentonite clay.

85 lbs. of calcium carbonate sand, 10 lbs. of Tennessee ball clay, 5 lbs. of bentonite clay and 0.5 lb. of corn starch were mixed together with a slight amount of water to form a paste. This mixture was fed into the hopper of a Chamber Bros. laboratory extruding machine. The mixture was extruded into a cylindrically shaped briquette 1½ inches in diameter and approximately 4 inches long. The briquette was then cut into four 1 inch pellets such that each pellet measured approximately 1 inch long × 1½ inches in diameter. The pellets were dried at 120° C for about 24 hours and then calcined at 1,370° C for 5 hours. While the pellets were still hot (426° C) they were immersed in a bath of molten magnesium and infiltrated. It was found that an average of 44 percent by weight of magnesium was infiltrated into the pellets.

EXAMPLE 6

This example illustrates the use of water glass (Na₂SiO₃ · 2H₂O) as a ceramic binder instead of bentonite clay as given in Example 1.

The procedure of Example 1 was followed using 1 percent water glass instead of 5 percent bentonite clay as a binder. 100 gms of oolitic sand were mixed with 1 gm of water glass. The mixture was formed into a briquette and the briquette was calcined at 1,100° C. The fired briquette weighed 66.9 gms after calcination. The briquette was then immersed in molten magnesium metal and the briquette weighed 133.4 gms after infiltration. After infiltration, it was found that 49.9 percent magnesium had been infiltrated into the briquette.

EXAMPLES 7 - 8

These examples illustrate the production of calcium carbonate sand briquettes containing 5 percent bentonite clay infiltrated with alloys of magnesium.

The procedure of Example 1 was followed except that two alloys of magnesium in the molten state were used to infiltrate two separate briquettes instead of substantially pure magnesium as in Example 1. The first alloy was a Mg-Ca alloy containing 5 percent calcium and the remainder magnesium; the second was a Mg-Ca-Li alloys containing 5 percent calcium, 2 percent lithium and the remainder magnesium. After weighing the briquettes before and after infiltration in these alloys, it was found that 59.0 percent by weight of the Mg-Ca alloy had been infiltrated into the first briquette and 59.6 percent of the Mg-Ca-Li alloy had been infiltrated into the second briquette.

Both of these alloy infiltrated briquettes were useful in desulfurizing a molten iron melt.

The operational details and the results obtained of all of these examples are recorded in the following table.

It should be noted that all of these briquettes were infiltrated with an amount of magnesium or magnesium alloys from 44 percent to 66 percent. All of these infiltrated briquettes are useful for desulfurizing molten metal in an efficient manner without forming a violent reaction.

While this invention has been described and illustrated by the examples shown, it is not intended to be strictly limited thereto, and other variations and modifications may be employed within the scope of the following claims.

2. Composition according to claim 1 in which there are from about 2 to about 50 parts by weight of the alkaline earth metal oxide for each part of binder.

3. Composition according to claim 1 in which the alkaline earth metal oxide is calcium oxide.

4. Composition according to claim 1 in which the porous refractory body composition is impregnated with an alloy of magnesium.

5. A porous compressed refractory body composition comprising a porous structure of an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, said structure containing magnesium absorbed in the interstices of said porous structure, the alkaline earth metal oxide having an average particle size below 4 mesh, the amount of said alkaline earth metal oxide being present in amount from about 30 percent to about 50 percent, said binder being present in amount from about 1 percent to about 15 percent and said composition being impregnated with at least about 35 percent magnesium absorbed in said structure, all of the percentages expressed on a weight basis.

6. A process for producing a porous, compressed refractory body comprising a porous structure of an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water, glass, cement and mixtures thereof, said structure containing magnesium absorbed in the interstices of said porous structure, said process which comprises admixing a particulate alkaline earth metal carbonate and a ceramic binder, the amount of said alkaline earth metal

TABLE

EXAMPLE	PRIOR TO CALCINATION			BRIQUETTING PRESSURE	CALCINING TEMPERATURE	% MAG. INFILTRATED
	% CaCO ₃ SAND	BINDER	% BINDER			
1	95.0%	Bentonite	5.0%	7500 psi	1100°C	47%
2	97.65%	Bentonite	2.35%	1500 psi	900°C	53%
3	97.65%	Bentonite	2.35%	7500 psi	900°C	52.4%
4	86.6%	Bentonite & Sawdust	2.40%	1500 psi	900°C	66%
5	85.0%	Tennessee Ball Clay	11.0%	—	1370°C	44%
		Bentonite	5%			
6	95.0%	Corn Starch	0.5%	7500 psi	1100°C	49.9%
		Water Glass	1%			% Mg-Ca ALLOY INFILTRATED
7	95.0%	Bentonite	5%	7500 psi	1100°C	59.0%
						% Mg-Ca-Li ALLOY INFILTRATED
8	95.0%	Bentonite	5%	7500 psi	1100°C	59.6%

We claim:

1. A porous compressed refractory body composition comprising a porous structure of an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, said structure containing magnesium absorbed in interstices of said porous structure, said refractory body composition having at least about 2 parts by weight of alkaline earth metal oxide present in said composition for each part of binder, said alkaline earth metal oxide having an average particle size below 4 mesh, and said composition being impregnated with at least 35 percent by weight of magnesium absorbed in said structure based on the total weight of the impregnated composition.

carbonate being present in amount from about 75 percent to about 99 percent, said ceramic binder being present in amount from about 1 percent to about 25 percent, all of the percentages expressed on a weight basis, forming pellets of said mixture, drying and firing said pellets at a temperature sufficient to convert the metal carbonate to metal oxide and to volatilize the carbon dioxide formed, thus forming a porous, compressed structure of a ceramic body, and impregnating the pores of said body with magnesium.

7. A process for producing a porous, compressed refractory body comprising a porous structure of an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, said structure containing

magnesium absorbed in the interstices of said porous structure, said process which comprises admixing a particulate alkaline earth metal carbonate and a ceramic binder, said alkaline earth metal carbonate being present in amount from about 75 percent to about 99 percent, said ceramic binder being present in amount from about 1 percent to about 25 percent, all of the percentages expressed on a weight basis, adding to said mixture sufficient water to form a moldable mixture, forming pellets from said mixture, drying and firing said pellets at a temperature from about 875° C to about 1,450° C to convert the metal carbonate to metal oxide and to volatilize the carbon dioxide formed, thus forming a porous, compressed and open structure of a ceramic body, immersing said body with magnesium and removing said impregnated body from said molten metal.

8. Process according to claim 7 in which the moldable mixture is compressed at a pressure from about 1,500 psi to about 30,000 psi to form pellets.

9. Process according to claim 7 in which the moldable mixture is extruded to form pellets.

10. Process according to claim 7 in which the alkaline earth metal carbonate is calcium carbonate.

11. Process according to claim 7 in which the alkaline earth metal carbonate is of a size of -4 mesh.

12. A process for producing a porous, refractory body comprising a porous structure of an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, said structure containing magnesium absorbed in the interstices of said porous structure, said process which comprises admixing a particulate alkaline earth metal carbonate, a ceramic binder and a cellulosic material, the amount of said alkaline earth metal carbonate being present in amount from about 55 percent to about 70 percent, said cellulosic material being present in amount from 10 percent to 30 percent, all of the percentages expressed on a weight basis, adding to said mixture from about 2 percent to about 6 percent water to form a moldable mixture, forming pellets from said mixture, drying and firing said pellets at a temperature from about 875° C to about 1,450° C to volatilize the carbon dioxide formed and to burn off the cellulosic material, thus forming pellets of a porous ceramic body having an open structure comprising said alkaline earth metal oxide and said binder, immersing said body in molten magnesium metal to fill the pores of said structure with magnesium and removing said impregnated body from said molten metal.

13. Process according to claim 12 in which the size

of the cellulosic material employed is -20 mesh.

14. Process according to claim 12 in which the cellulosic material is selected from the group consisting of sawdust, corn cob grits, corn stalks and oat hulls, and mixtures thereof.

15. A process for treating molten iron which comprises immersing a porous structure comprising a refractory body composition impregnated with magnesium into said molten iron to reduce the sulfur content thereof, the porous refractory body composition comprising an alkaline earth metal oxide and a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, said refractory binder having from about 2 to about 50 parts by weight of alkaline earth metal oxide for each part of binder, the alkaline earth metal oxide present in said composition having an average particle size below 4 mesh, and said composition being impregnated into the pores of said structure with at least about 35 percent by weight of magnesium.

16. A process for treating molten iron which comprises immersing a porous structure comprising a refractory body impregnated with magnesium into said molten iron to reduce the sulfur content thereof, said porous refractory body comprising from about 30 percent to about 50 percent alkaline earth metal oxide, from about 1 percent to about 15 percent of a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, and said body impregnated into the pores of said structure with from about 35 percent to about 70 percent magnesium, all of the percentages expressed on a weight basis.

17. Process according to claim 16 in which the magnesium employed is a magnesium alloy.

18. A process for desulfurizing molten iron which comprises immersing a porous structure comprising a refractory body impregnated with magnesium into said molten iron for a period of about 1 to about 10 minutes and removing said body from said molten iron after said period, said refractory body comprising from about 30 percent to about 50 percent alkaline earth metal oxide, from about 1 percent to about 15 percent of a ceramic binder, said binder selected from the group consisting of clay, water glass, cement and mixtures thereof, and said body impregnated into the pores of said structure with from about 35 percent to about 70 percent magnesium, all of the percentages expressed on a weight basis.

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