

CONVENTION

AUSTRALIA
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596861

77196/87

APPLICATION FOR A STANDARD PATENT

I/We The Dow Chemical Company

NO OBJECTION ACCEPTED AND AMENDMENTS

of 2030 Dow Center,
Abbott Road,
Midland,
Michigan 48640,
UNITED STATES OF AMERICA.

ALLOWED 27-2-90

hereby apply for the grant of a standard patent for an invention entitled:

INJECTABLE REAGENTS FOR MOLTEN METALS

which is described in the accompanying complete specification.

Details of basic application

Number of basic application: 899,859

Convention country in which
basic application was filed: UNITED STATES OF AMERICA

Date of basic application : 25 August 1986

Address for Service:

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MAIL OFFICER: [Signature]

Dated: 24 July 1987

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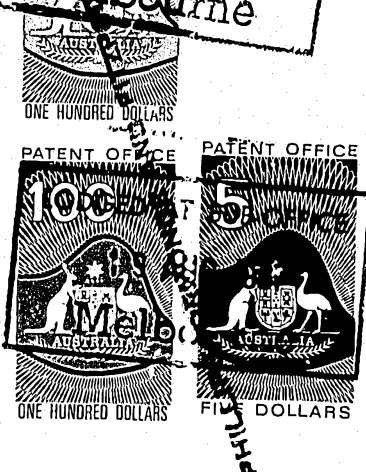
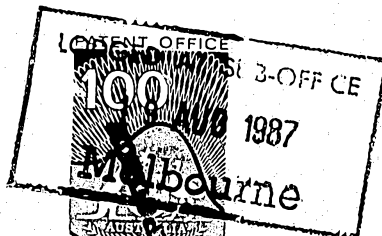
By:

David B Fitzpatrick

Our Ref: 62310
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Melbourne

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DECLARATION FOR A PATENT APPLICATION

INSTRUCTIONS

- (a) Insert "Convention" if applicable
- (b) Insert FULL name(s) of applicant(s)
- (c) Insert "of addition" if applicable
- (d) Insert TITLE of invention

In support of the (a) Convention application made by
 (b) THE DOW CHEMICAL COMPANY
 2030 Dow Center, Abbott Road,
 Midland, Michigan 48640, UNITED STATES OF AMERICA
 (hereinafter called "applicant(s)" for a patent (c) for an
 invention entitled (d) INJECTABLE REAGENTS FOR MOLTEN METALS

- (e) Insert FULL name(s) AND address(es) of declarant(s) (See heading*)

I/We (e) Richard G. Waterman, General Patent Counsel
 THE DOW CHEMICAL COMPANY
 2030 Dow Center, Abbott Road,
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 do solemnly and sincerely declare as follows:

- 1. ~~I am/We are the applicant(s).~~
 (or, in the case of an application by a body corporate)
- 1. I am/We are authorized to make this declaration on behalf of the applicant(s).
- 2. ~~I am/We are the actual inventor(s) of the invention.~~
 (or, where the applicant(s) is/are not the actual inventor(s))
- 2. EDWARD J. SKACH, JR., 816 N. Peach Point, P.O.Box 2972, Freeport, Texas 77541; JOSEPH H. WAIBEL, 336 Linden Lane, Lake Jackson, Texas 77566; DAVID W. BRAKE, 1411 Rev. Swinney Street, West Columbia, Texas 77486; all of the United States of America
 is/are the actual inventor(s) of the invention and the facts upon which the applicant(s) is/are entitled to make the application are as follows:
- (8) The applicant Company is the assignee of the said invention from the said actual inventor(s).

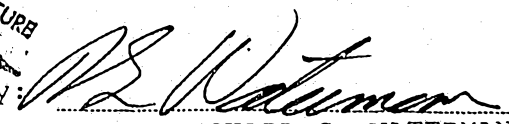
(Note: Paragraphs 3 and 4 apply only to Convention applications)

- 3. The basic application(s) for patent or similar protection on which the application is based is/are identified by country, filing date, and basic applicant(s) as follows:
 (a) U.S.A. Patent Application Serial No. 899,859 which was filed August 25, 1986 in the names of Edward J. Skach, Jr., Joseph H. Waibel and David W. Brake
- 4. The basic application(s) referred to in paragraph 3 hereof was/were the first application(s) made in a Convention country in respect of the invention the subject of the application.

- (k) Insert PLACE of signing
- (l) Insert DATE of signing
- (m) Signature(s) of declarant(s)

Declared at (k) Midland, Michigan, 48640,
 Dated (l) July 2 1987 U.S.A.
 (m) THE DOW CHEMICAL COMPANY

CORP.
SEAL

SIGNATURE
 BY: 
 RICHARD G. WATERMAN
 General Patent Counsel

To: The Commissioner of Patents

Agent:

(12) PATENT ABRIDGMENT (11) Document No. AU-B-77196/87
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(54) Title
MAGNESIUM/ALUMINIUM IMPREGNATED INJECTABLE ADDITIVE FOR IRON & STEEL

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(56) Prior Art Documents
EP 16273
US 3801303

(57) Claim

1. A process for preparing an injectable reagent for a molten process metal, comprising the steps of:

impregnating a minor portion of a metal reagent, in molten form, into a major portion of non-molten particles of a particulate inorganic reagent, in an atmosphere that is substantially devoid of extraneous reactants,

cooling the so-impregnated particles to freeze the metal reagent, and

recovering the particulate inorganic reagent having metal reagent impregnated therein, said metal reagent comprising less than 50 percent by weight of the particles.

2. The process of Claim 1, wherein the metal reagent is selected from magnesium aluminum, and alloys thereof, and wherein the metal reagent comprises from 5 percent to 40 percent by weight of the total weight of

the injectable reagent.

4. The process of Claim 1, 2, or 3, wherein the inorganic reagent is an aluminum compound or an alkaline earth metal compound.

5. The process of Claim 1, 2, or 3, wherein the inorganic reagent is selected from CaO , CaC_2 , MgO , CaAl_2O_4 , ~~Al_2O_3~~ and mixtures of these or Al_2O_3 .

8. A process for preparing an injectable reagent for a molten process metal, said molten process metal having a melting point greater than ~~about~~ 950°C , comprising the steps of:

mixing together a molten reagent metal selected from aluminum, magnesium, and alloys thereof with at least one particulate non-molten inorganic reagent compound of at least one metal selected from Ca, Mg, and Al,

conducting said mixing in an environment which is substantially devoid of extraneous reactants and for a time sufficient to obtain a substantially uniform distribution of the molten metal reagent in the said inorganic reagent compound, thereby effecting permeation of the molten reagent metal into the particles of inorganic reagent compound,

cooling the mixture to below the freezing point of the molten reagent metal, and

recovering the particulate inorganic reagent compound permeated with said reagent metal, said metal reagent comprising less than 50 percent by weight of the particles.

9. An injectable reagent for a molten process metal, said reagent comprising:

a particulate inorganic reagent the particles of which are impregnated with a metal reagent, wherein the metal reagent comprises less than 50 percent by weight of the particles.

INJECTABLE REAGENTS FOR MOLTEN METALS

The invention relates to magnesium-containing and/or aluminum-containing injectable reagents for molten metals.

5 Magnesium (Mg) is well known as an injectable for molten metals. In some cases Mg is used as an alloying agent, or as a deoxidizer, or as a desulfurizer and, in some cases, as a nodularizer. Aluminum (Al) has also been used as an injectable for
10 molten metals, especially as an aid for a calcium compound, e.g. lime (CaO), which is used as a desulfurizing agent for molten iron. Calcium (Ca) may be used in place of Mg, but it is not cost-competitive
15 with Mg or Al.

20 It is known that Mg powder or Al powder can be used along with a calcium compound, e.g. CaO, by being injected into molten iron either as a physical mixture with a particulate Ca compound or by staged successive injections of the Mg or Al with the Ca compound. Additional publications representative of the state of
25 the art are the following:

U.S. Patent No. 4,137,072 discloses a molded pellet of a mixture of at least one metal selected from Mg, Ca, and Al and at least one metal oxide selected from MgO, CaO, and Al₂O₃. Preference for Mg + MgO is shown. The use of an organic polymer binding material as an optional ingredient in the mixture is disclosed.

5 U.S. Patent No. 4,139,369 discloses a mixture of Mg powder with CaO, CaCO₃, CaC₂, or CaMg(CO₃)₂, wherein the Ca compound has a particle size of from 0.06 to 3 mm and the Mg particles have a size of from 0.060 to 0.095 mm.

10 U.S. Patent No. 4,173,466 discloses compacted tablets of particulate magnesium, calcium, and iron in which the iron is the predominant ingredient.

15 U.S. Patent No. 4,182,626 discloses a staged mixing process for combining pulverulent Mg metal with fine particle alkaline earth metal compounds.

20 U.S. Patent No. 4,209,325 discloses a mixture of alkaline earth metal with sintered CaO which contains at least one fluxing agent, said fluxing agent

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being, e.g. alumina, alkali metal fluoride, alkaline earth metal fluoride, or sodium carbonate.

U.S. Patent No. 4,586,955 discloses the use of Al powder with CaO to desulfurize hot metal in a ladle.

U.S. Patent Nos. 4,559,084 and 4,421,551 disclose salt-coated Mg granules for use in desulfurizing molten iron.

In spite of general successes which have been achieved in using Mg or Al particles along with such materials as CaO and CaC₂ as an injectable in molten process metals, e.g. molten iron, there remains a need in the industry for an injectable which does not create excessive, unwanted splashing of the molten process metal as the injectable is undergoing a reaction therein, which is uniform in composition, which is more easily and safely handled, and which is non-segregating during shipping, storage, and handling.

The present invention particularly resides in a process for preparing an injectable reagent for a molten process metal, comprising the steps of:

impregnating a minor portion of a metal reagent, in molten form, into a major portion of non-molten particles of a particulate inorganic reagent, in an atmosphere that is substantially devoid of extraneous reactants,

cooling the so-impregnated particles to freeze the metal reagent, and

recovering the particulate inorganic reagent having metal reagent impregnated therein, said metal

reagent comprising less than 50 percent by weight of the particles.

5 The invention further resides in a process for preparing an injectable reagent for a molten process metal, said molten process metal having a melting point greater than ~~about~~ 950°C, comprising the steps of:

10 mixing together a molten reagent metal selected from aluminum, magnesium, and alloys thereof with at least one particulate non-molten inorganic reagent compound of at least one metal selected from Ca, Mg, and Al,

15 conducting said mixing in an environment which is substantially devoid of extraneous reactants and for a time sufficient to obtain a substantially uniform distribution of the molten metal reagent in the said inorganic reagent compound, thereby effecting permeation of the molten reagent metal into the particles of inorganic reagent compound,

20 cooling the mixture to below the freezing point of the molten reagent metal, and

25 recovering the particulate inorganic reagent compound permeated with said reagent metal, said metal reagent comprising less than 50 percent by weight of the particles.

30 The invention additionally resides in an injectable reagent for a molten process metal, said reagent comprising

35 a particulate inorganic reagent the particles of which are impregnated with a metal reagent, wherein the metal reagent comprises less than 50 percent by weight of the particles.



The injectable reagents of the present invention are prepared by effecting a permeation of molten Mg or Al, or alloys thereof (i.e. metal reagents) into at least one particulate, non-molten, inorganic, alkaline earth metal reagent compound such as CaO, CaC₂, MgO, CaAl₂O₄, dolime or mixtures of these, or, e.g., Al₂O₃, and the like, under conditions wherein the amount of the molten metal reagent does not exceed the amount of the particulate inorganic reagent material. A substantially inert atmosphere, esp. argon, is maintained in some instances as long as the Mg or Al is hot to prevent reaction with air.

One process for effecting the permeation of the molten metal reagent into the particulate, inorganic reagent material comprises physically adding the molten metal reagent, preferably with stirring, to the inorganic reagent material whereby the molten metal reagent permeates the inorganic reagent particles. Another process comprises blending particulate Mg or Al, or alloys thereof, (i.e. metal reagent) with the particulate inorganic reagent material in the desired ratios, then heating the mixture to melt the metal reagent, thereby effecting permeation of the molten metal reagent into the non-molten inorganic reagent material.

After the molten metal reagent has permeated the particulate inorganic reagent material, the mixture is cooled to freeze the metal in place in the inorganic particles. If any of the particles are adhered together by the presence of metal reagent on the surface of the particles, one may, if desired, grind

the particles to obtain a free-flowing particulate product.

5 The injectable of this invention is characterized as a particulate inorganic material (more fully described hereinafter) impregnated with Mg or Al or alloys thereof, in which the particulate inorganic material comprises a major proportion of the total weight of the injectable.

10 For purposes of conciseness and ease of description the following terminology is used:

15 1. The term "metal reagent" herein refers to Mg, Al, or alloys of these, employed in the "injectable reagent";

20 2. The term "particulate inorganic reagent" herein refers to ^{alkaline} earth metal compound(s) and/or aluminum compound(s) into which the "metal reagent" is impregnated;

25 3. The term "impregnated" herein is used synonymously with "permeated" and "imbibed" to indicate a "soaking-in" effect;

30 4. The term "injectable" or "injectable reagent" refers to the "particulate inorganic reagent" into which the "metal reagent" has been impregnated, the "injectable" is particularly useful as an injectable for molten process metal and is a composite of the metal reagent and the inorganic reagent;

35 5. The term "process metal" is the metal into which the injectable is injected or introduced.



The injectable of the invention comprises a particulate inorganic reagent impregnated with the metal reagent, said impregnation having been effected by an imbibing or permeating of the molten metal reagent into the particulate inorganic reagent, followed by freezing of the metal reagent. Impregnation is effected, in some instances, in an environment which is substantially devoid of an extraneous reactive atmosphere, such as air (oxygen, moisture). Argon is preferred as a non-reactive, inert atmosphere and is readily available. CaC_2 does not appear to promote the oxidation of the metal reagent, thus when the only inorganic reagent being used is CaC_2 , then argon is not usually necessary and ambient air can be used, but too much moisture can be detrimental and is best avoided.

The impregnation of the metal reagent may be effected by mixing the metal reagent in a molten condition into the non-molten particulate inorganic reagent, followed by freezing and, if needed, by breaking up clusters of adhered particles and by grinding to obtain the preferred particulate form. For best results, the inorganic reagent should be pre-heated to above the melting point of the metal reagent so as to avoid having the molten metal reagent freeze before being well-mixed.

It is generally preferred, however, that impregnation of the metal reagent into the particulate inorganic reagent be effected by blending particulate metal reagent with the particulate inorganic reagent, then heating to a temperature sufficient to melt the metal reagent, but not the inorganic reagent, thereby effecting impregnation of the metal reagent into the

inorganic reagent, then cooling to freeze the metal:
Here too, an extraneous reactive atmosphere such as
air, especially while the metal reagent is hot, should,
in some instances, be avoided by using an inert
5 atmosphere such as argon. Some regrinding may be
needed to break up clusters of particles.

Each of the above-described processes for
making the injectable has features which may commend it
10 in certain circumstances. For instance, if it is
desired to use a metal reagent which is already
available in molten form, such as at the point of
manufacture of the metal or its alloy, one may
economize by mixing the already molten metal into the
15 particulate inorganic reagent. However, if the
available metal is not already molten, it is preferred
to mix the non-molten metal in particulate form with
the particulate inorganic reagent prior to melting of
the metal to obtain the impregnation. By this method a
20 substantially uniform mixture is obtained prior to
melting of the metal and the time required at molten
temperature is shortened, thus economizing on energy.
Furthermore, by pre-mixing the reagents in the solid,
25 particulate form, a more homogeneous product can be
obtained, especially throughout a large batch.

If one mixes a small amount of the molten metal
reagent into a batch of the particulate inorganic
30 reagent, then some of the inorganic particles are
likely to be thoroughly permeated by the molten metal
while other particles may imbibe little or none of the
molten metal. The particles, when well-mixed, will
35 comprise a uniform blend, but not a homogeneous batch.

A uniform blend of particles is operable, but a homogeneous batch of particles is preferred.

5 The metal reagents, are selected from Mg, Al, or alloys of these, especially Mg or its alloys thereof.

10 The particulate inorganic reagents comprise alkaline earth metal compounds and/or aluminum compounds which are substantially stable during the process of preparing the injectable of this invention. There may be some reaction during the process if any
15 extraneous reactants, such as air or moisture, accompany the inorganic reagent and for this reason it is preferable to pre-heat the inorganic reagent compound prior to combining it with the metal reagent, thus driving out air, moisture, or other volatile
20 extraneous material which can react with the metal reagent and reduce the effectiveness of the metal reagent. It is desirable to retain the reactivity effectiveness of the metal reagent throughout the formation of the injectable so that the reactivity of the metal reagent desired during the molten process
25 metal treatment is preserved until that time. Thus, during the permeation of the metal reagent into the particles of inorganic reagent it is desired that there be little or no chemical reaction occurring, other than release of whatever volatile materials that may be
30 present. If one is aware, however, that some air (oxygen) or moisture may be present, one may add enough excess of the metal reagent to allow for some of it to be converted to MgO or Al₂O₃, thus increasing the amount of the inorganic reagent.
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Mixing of the particulate metal reagent and the particulate inorganic reagent may be done in mixers designed to provide mixing of dissimilar solid particles or mixing a liquid with a particulate solid.

5 Heated rotating vessels may be used which are operated at a temperature high enough to melt the metal reagent. It is within the skill of practitioners of the art of such mixing to select, or design, vessels of a type and size suitable for their particular needs including, if
10 needed, the provision of an inert gas pad or purge in the vessel. Pre-mixing of metal particles and inorganic particles may be done at a temperature below the melting temperature of the metal particles after
15 which the reagents are conveyed into a heated mixer wherein the metal particles are melted, thereby effecting permeation of the molten metal into the inorganic particles.

20 The present injectable is variously useful in molten process metals (esp. those melting at a temperature above 950°C) which variously require deoxidation, desulfurization, graphite nodularization, or silicon-content modification. The injectable of the
25 invention is especially useful as deoxidation or desulfurization reagents for molten ferrous metals. The amount of the reagents required in a given application will be determined by the operator of the
30 process metal treating process.

35 The injectable of the invention, generally is in particulate form of a size small enough to fall through an 8-mesh (2.38 mm) screen (U.S. Standard Sieve Size), preferably through a 40-mesh (0.42 mm) screen, most preferably through a 100-mesh (0.149 mm) screen. The injectable may be injected into the molten process

metal through a lance in a carrier stream of inert gas (e.g., nitrogen or argon) or by the wire injection technique. The wire injection technique involves packing of the injectable into a small diameter hollow tube of a metal which dissolves in the process metal. For instance, for molten iron (or steel) desulfurization a thin-walled, small-diameter hollow tube of iron (or steel) filled with the injectable reagents would be inserted into the molten iron at a rate controlled by the operator of the process which has been determined as the rate needed to obtain the desired results. Injection by way of a lance or as a wire are well known in the metal processing industry and further explanation here should not be needed. Also the temperature at which the molten process metal should be processed is determined by the operator of the process.

The temperature at which the molten metal reagent is impregnated into the inorganic reagent must, of course, be at or above the melting point of the metal reagent and, preferably, should not very closely approach the boiling point which, because of the significant vapor pressure of the metal, can cause loss of the metal reagent into the atmosphere. The melting point of Mg is about 650°C and the melting point of Al is about 660°C. The boiling points of the various alloys of Mg and Al are found in various texts and publications. It has been found that a temperature range of from 800° to 900°C provides a low viscosity, but not too vaporous, Mg.

In the case of Mg, for example, if used in a manner in which the Mg is the major ingredient in contact with a portion of CaO reagent, such as when

molten Mg is added to a batch of CaO reagent, the temperature must be kept above 715°C to avoid the formation of a Mg-Ca alloy. So long as the injectable reagent is prepared in such a manner that the CaO reagent is the major ingredient and the molten Mg is the minor ingredient, then the formation of a Mg-Ca alloy is avoided, even at molten Mg temperatures below 715°C. Thus, the CaO comprises a major amount (more than 50 percent) and the Mg comprises a minor amount (less than 50 percent) of the total weight of the injectable reagent; preferably the Mg is an amount in the range of about 5 percent to ~~about~~ 40 percent, most preferably about 25 percent to ~~about~~ 35 percent.

The impregnation of molten metal reagent into the inorganic reagent works better with high porosity particles than with low porosity (i.e. densified) particles. Particles of Al_2O_3 , for example, tend to become densified (less porous) when heated at very high temperatures, particularly ~~above~~ 1000°C to 1200°C; these densified particles, being almost like a ceramic material may not imbibe much of the molten metal, so the metal collects mainly on the surface of the particles.

Injectables made in accordance with the present invention offer distinct advantages over the use of mere physical mixtures of metal particles and inorganic particles, e.g., lack of segregation during storage, handling, shipping, more efficient desulfurizing, better batch-to-batch conformity, and fewer off-spec process metals.

The following examples illustrate various embodiments, but the present invention is not limited



to the particular embodiments illustrated. In the examples, all "parts" and percentages are by weight unless noted otherwise.

5 Example 1: Mg-Impregnated CaO

10 In an argon atmosphere 20 parts by weight of Mg particle size powder is uniformly blended with 40 parts by weight of dry calcined particle size CaO. The blended mixture is placed in a ceramic vessel and placed in an argon-purged oven preheated to a temperature of 800°C to 900°C and heated for about 10 minutes, thereby melting the Mg powder where-upon it "soaks" into the CaO particles. The particles are removed from the oven and cooled in an argon atmosphere. At ambient temperature the particles are easily handled in ambient air; clusters of particles are easily broken apart as demonstrated by using a mortar and pestle. Because of having been uniformly blended prior to the melting of the Mg powder, the product is substantially homogeneous, that is, each particle is substantially of the same composition as the other particles. By having the oven preheated to a temperature of 800°C to 900°C, melting of the Mg metal powder takes place more rapidly than if the oven is operated at a lower temperature of, for example, 650°C to 750°C.

30 An operable, but less homogeneous product is prepared in a process wherein molten Mg is added, with mixing, into the calcined CaO, because the molten Mg soaks into some of the CaO particles before there is an opportunity for it to be uniformly distributed throughout the particles and an extensive mixing period is

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needed to more thoroughly distribute the molten Mg so as to more closely approach homogeneity.

5 Furthermore, if one adds CaO particles into molten Mg metal, then at the outset at least, there is, in the mixing vessel, a large excess of molten Mg for the amount of CaO added. This tends to cause the CaO and Mg to react and form a significant amount of MgO and Mg-Ca alloy. The present invention effectively
10 avoids that by using techniques wherein the amount of CaO is always greater than the amount of Mg.

Example 2: Mg-Impregnated CaC_2

15 In an ambient laboratory atmosphere 20 parts of Mg powder is uniformly blended with 40 parts of CaC_2 (-300 mesh). The blend is placed into an oven preheated to 800°C to 900°C thereby melting the Mg whereupon it is imbibed by the CaC_2 . After about ten
20 minutes it is removed from the oven and cooled. Any clusters or bumps formed are easily broken into a powder using a mortar and pestle.

25 It is notable that when CaC_2 is used and oxide-containing reagents are not used, the need for an argon purge to exclude air is substantially obviated, because oxides (e.g. CaO) tend to promote the oxidation of Mg metal (when hot or molten), thus upsetting the targeted
30 amount of Mg in the final injectable reagent, giving rise to increased amounts of MgO. However, any MgO present in the mixture can function as part of the inorganic reagent portion of the injectable reagent.

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Example 3: Mg-Impregnated CaC_2

In accordance with the method of Example 2, 20 parts of Mg powder are used with 60 parts of the particulate CaC_2 to make an injectable. The results are essentially the same except that here the Mg comprises about 25 percent of the total as compared to about 33 percent of the total in Example 2.

Example 4: Mg-Impregnated MgO

In accordance with the procedure followed in Example 1 above, about 12 parts of Mg powder are used with about 35 parts of particulate MgO , using an argon atmosphere to produce an injectable reagent. When cooled to room temperature the product can be handled in regular ambient air. Clusters are broken up with a mortar and pestle.

Example 5: Mg -Impregnated CaAl_2O_4

Following the procedure of Example 2, using "regular" ambient air as the atmosphere, about 27 parts of Mg powder and about 80 parts of particulate calcium aluminate, CaAl_2O_4 , are used in making an injectable reagent. Clusters are broken up with a mortar and pestle.

Example 6: Mg-Impregnated Al_2O_3

Following the procedure of Example 2, using ambient atmosphere, 30 parts of Mg powder and 90 parts of Al_2O_3 powder are used in making an injectable reagent. Product is easily handled in regular ambient air. Clusters are more difficult to break apart, apparently because the Al_2O_3 had very little porosity



and much of the Mg was on the surface of the particles, adhering them.

Example 7: Mg-Impregnated CaO/MgO (Dolime)

5 Following the procedure of Example 1, in argon, 20 parts of Mg powder and 60 parts of particulate dolime are used in making an injectable reagent. When cooled to room temperature the product can be handled in regular ambient air. Clusters are broken up with a mortar and pestle.

Example 8: Mg-Impregnated CaO/CaC₂

15 Following the procedure of Example 1, in argon, 20 parts of Mg powder, 30 parts of dry-calcined, particulate CaO, and 30 parts of particulate CaC₂ (-300 mesh) are used in making an injectable. The powder can be handled in regular air. Clusters are easily broken up using a mortar and pestle.

Example 9: Mg-Impregnated CaO

25 This demonstrates that the present methods and products are not seriously affected by the presence of minor amounts of extraneous salts or other such ingredients. The particulate Mg used here is a salt-coated Mg granule such as is disclosed in U.S. Patent No. 4,559,084. The salt-coated Mg granules (about 92 weight percent Mg) are of a size which will all pass through an 8 mesh screen.

30 Following the procedure of Example 1, in argon, 20 parts of the salt-coated Mg granules are used with about 60 parts of dry-calcined particulate CaO to successfully prepare an injectable reagent. The

presence of the salt (mostly alkali metal halides) has no substantial effect on the operation of the process or on the use of the injectable reagent in treating a molten process metal. Too much salt can, however, increase the amount of splashing of the process metal.

Example 10: Mg-Impregnated CaC_2

In the same manner as Example 9, but in regular air, 20 parts of the salt-coated Mg granules are successfully used with 60 parts of CaC_2 . (-300 mesh) to make an injectable.

Example 11: Mg-Impregnated CaO/CaC_2

In the same manner as Example 9, using argon, 20 parts of the salt-coated Mg granules are successfully used with 30 parts of CaO and 30 parts of CaC_2 to make an injectable.

Example 12: Mg-Impregnated CaO

This example demonstrates that the Mg may be in a form other than powder or granules.

In the same manner of Example 1, under argon, 20 parts of particulate Mg scrap (lathe shavings) are successfully used with 60 parts of dry-calcined particulate CaO to make an injectable reagent.

Example 13: Mg-Impregnated CaO/CaC₂

In the same manner as Example 12, 20 parts of Mg shavings are successfully used with 30 parts of CaO and 30 parts of CaC₂ to make an injectable reagent.

Example 14: Comparison With Prior Art Mg/CaO Blend

In a small-scale demonstration, comparative runs were made between injectable reagents of the present invention and a physical mixture of Mg powder/-lime powder, representing commonly used prior art. The runs were performed by injecting the reagents through a lance, in a nitrogen gas, into a small ladle of molten steel. All 3 runs had about equal violence.

	<u>Reagent</u>	<u>% Mg in Reagent</u>	<u>Relative¹ Desulfurization</u>
20	Mg/CaO blend ²	30	45
	M.I.L. ³	30	60
	M.I.C.C. ⁴	30	90

¹Relative efficiency of the desulfurization.

²An example of prior art blend of Mg powder and lime.

³Magnesium-impregnated-lime.

⁴Magnesium-impregnated-calcium carbide.

As shown above, the M.I.L. is almost 50 percent more efficient than the prior art blend as a desulfurizing reagent. M.I.C.C. is twice as efficient. Further, the M.I.L. and M.I.C.C. do not undergo segregation of Ca compound from the Mg metal during storage, shipping, or handling.

Example 15: Comparison With Prior Art Mg/CaO Blend

5 In a large scale commercial-size ladle, using lance injection in nitrogen, a large number of tests were made so as to compare M.I.L. (Mg-impregnated-lime of the present invention) with a prior art blend of Mg/CaO. The reagents each contained about 25 percent by weight Mg. The average desulfurization efficiency was determined, along with the standard deviation.

10

<u>Reagent</u>	<u>Efficiency</u>	<u>Std. Deviation</u>
Mg/CaO blend (prior art)	39	19
M.I.L. (present invention)	61	9

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20 This compares extremely well with the efficiency results found in the small-scale demonstrations of the above example. The M.I.L. was about 50 percent more efficient than the prior art blend and had much less deviation between runs.

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The claims defining the invention are as follows:

1. A process for preparing an injectable reagent for a molten process metal, comprising the steps of:

5 impregnating a minor portion of a metal reagent, in molten form, into a major portion of non-molten particles of a particulate inorganic reagent, in an atmosphere that is substantially devoid of extraneous reactants,

 cooling the so-impregnated particles to freeze the metal reagent, and

10 recovering the particulate inorganic reagent having metal reagent impregnated therein, said metal reagent comprising less than 50 percent by weight of the particles.

15 2. The process of Claim 1, wherein the metal reagent is selected from magnesium aluminum, and alloys thereof, and wherein the metal reagent comprises from 5 percent to 40 percent by weight of the total weight of the injectable reagent.

20 3. The process of Claim 1 or 2, wherein the metal reagent comprises from 25 percent to 35 percent by weight of the total weight of the injectable reagent.

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4. The process of Claim 1, 2, or 3, wherein the inorganic reagent is an aluminum compound or an alkaline earth metal compound.

5 5. The process of Claim 1, 2, or 3, wherein the inorganic reagent is selected from CaO, CaC₂, MgO, CaAl₂O₄, ~~Al₂O₃~~ and mixtures of these or Al₂O₃.

10 6. The process of any one of the preceding claims wherein the impregnation of a the metal reagent into the particulate inorganic reagent is achieved by blending the particulate metal reagent with the inorganic reagent, and then melting the metal reagent to thereby obtain a permeation of the molten metal
15 reagent into the particles of inorganic reagent.

20 7. The process of any one of Claims 1 to 5, wherein the impregnation of the metal reagent into the particulate inorganic reagent is achieved by adding, with mixing, the metal reagent in the molten state into the particulate inorganic reagent, thereby obtaining a permeation of the molten metal reagent into the particles of inorganic reagent.

25 8. A process for preparing an injectable reagent for a molten process metal, said molten process metal having a melting point greater than ~~about~~ 950°C, comprising the steps of:

30 mixing together a molten reagent metal selected from aluminum, magnesium, and alloys thereof with at least one particulate non-molten inorganic reagent compound of at least one metal selected from Ca, Mg, and Al,

35 conducting said mixing in an environment which is substantially devoid of extraneous reactants and for



a time sufficient to obtain a substantially uniform distribution of the molten metal reagent in the said inorganic reagent compound, thereby effecting permeation of the molten reagent metal into the particles of inorganic reagent compound,

cooling the mixture to below the freezing point of the molten reagent metal, and

recovering the particulate inorganic reagent compound permeated with said reagent metal, said metal reagent comprising less than 50 percent by weight of the particles.

9. An injectable reagent for a molten process metal, said reagent comprising:

a particulate inorganic reagent the particles of which are impregnated with a metal reagent, wherein the metal reagent comprises less than 50 percent by weight of the particles.

~~10. The injectable reagent of Claim 9, wherein the said metal reagent comprises from 5 percent to 45 percent of the total weight of the injectable reagent and wherein the metal reagent is selected from magnesium, aluminum and alloys of these.~~

11. The injectable reagent of Claim 9, or 10, wherein the inorganic reagent is an aluminum compound or an alkaline earth metal compound.

12. The injectable reagent of Claim 9, 10, or 11, wherein the inorganic reagent is selected from CaO, CaC₂, MgO, CaAl₂O₄, Al₂O₃, and mixtures of these.

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10. The injectable reagent of claim 9, wherein the said metal reagent comprises from 5 percent to 40 percent of the total weight of the injectable reagent and wherein the metal reagent is selected from magnesium, aluminum and alloys of these.

11. The injectable reagent of claim 9 or 10, wherein the inorganic reagent is an aluminum compound or an alkaline earth metal compound.

12. The injectable reagent of claim 9, 10 or 11, wherein the inorganic reagent is selected from CaO, CaC₂, MgO, CaAl₂O₄, and mixtures of these or Al₂O₃.

13. A process according to claim 1 substantially as hereinbefore described with reference to any one of the examples.

14. An injectable reagent according to claim 9 substantially as hereinbefore described with reference to any one of the examples.

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