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MAGNESIUM OXIDE COATING COMPOSITION AND PROCESS

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3,583,887 MAGNESIUM OXIDE COATING COMPOSITION AND PROCESS John F. Steger and Jay W. Palmer, Crystal Lake, Ill., assignors to Morton International, Inc., Chicago, Ill. Filed Aug. 18, 1969, Ser. No. 850,910 Int. Cl. B23b 15/04; H01f 1/04 U.S. Cl. 148-27 **15 Claims**

ABSTRACT OF THE DISCLOSURE

Magnesium oxide coating compositions and process for applying to silicon-containing magnetic steel an adherent coating which acts as a separating medium to prevent fusing or welding of coiled sheet during an anneal- 15 ing process and which coating provides electrical insulating and corrosion resistant properties. The coating compositions comprise a major proportion of magnesium oxide and a minor proportion of additives containing a boron compound, a sodium compound, and silica or a 20 silicon-containing compound.

BACKGROUND OF THE INVENTION

Field of the invention

Electrical transformers and other types of electrical induction apparatus are made using core materials made from steel sheet having soft magnetic properties. Such sheet is prepared by cold-rolling silicon-containing steel 30 into sheet form, coiling the steel sheet into rolls and thereafter annealing the coiled steel by a controlled heating process to produce a grain oriented structure having desirable magnetic properties.

Magnesium oxide is used extensively as a highly heat 35 resistant separator medium and protective coating for metal surfaces. It is also used as an electrical insulator coating for metals, as a "gatherer" for removing impurities, such as sulfur and carbon, from thin metal sheets and particularly as a protective coating for silicon steel. The electrical insulating coating is understood to be derived from coating magnesium oxide on steel, which then forms a film or coating containing Mg₂SiO₄ which coating is an effective electrical insulator when annealed.

Description of the prior art

According to the present industrial practice, siliconcontaining steel is cold rolled into sheets, decarburized and thereafter coiled into convenient rolls. Cold rolling develops in the steel the potential to form a grain oriented 50structure when the steel is later "annealed." The term annealed refers to a process whereby the steel is heated to about 1200° C. in an essentially pure hydrogen atmosphere (or vacuum) under programmed conditions with respect to time and temperature. This results in a growth 55 in size of the steel grains and also in a specific grain orientation which provides the desired soft magnetic properties sought. During the annealing process, virtually all of the remaining excess carbon and sulfur content of the steel is lost. This type of annealing is known in the art 60 as "box annealing."

The steel so produced is in the form of a long, thin, coiled sheet which must be uncoiled and thereafter fabricated into the desired shape and size for its intended use. In the process of fabrication which can include, for ex-65 ample, punching, stamping and bending, strains and stresses are introduced in the steel which impair its magnetic properties. Consequently, the strains and stress must be relieved. This is accomplished by a process termed stress relief annealing which comprises reheating the fabricated steel to a temperature of about 800° C. in a 70 reducing atmosphere for a specified period of time.

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During the box annealing process where the steel is in large coils, in the absence of a suitable separating medium, the coiled roll would fuse to itself and could not be unrolled. Conventionally, this is avoided by placing a thin coating of magnesium oxide on the steel prior to coiling. Further, the magnesium oxide coating serves to reduce impurities such as carbon and sulfur in steel by chemical reaction. In addition, magnesium oxide provides an insulating silicate layer by reaction with the silicon 10 in the steel. There is also evidence that some iron silicate is formed. For certain applications, it is also important to form an efficient electrical insulating coating. Thus, for example, transformer cores are constructed from thin sheets of soft magnetic steel stacked together to form a laminated body in which each sheet is electrically insulated from its neighbor. This construction vastly reduces the generation of eddy currents in the core imposed by an alternating electrical field. The average density of soft iron in the core should be as large as possible and consequently the insulation on the plates should be as thin as possible to provide closer stacking of steel plates.

Obviously, short circuits between plates reduces transformer efficiency and often causes the development of damaging hot spots in the transformer core. Consequently soft magnetic steel is rated by the number of short circuits per unit area, usually expressed in terms of the electrical resistance of the insulating layer. This is a standard ASTM measurement known as the Franklin Test.

Since stress relief annealing is sometimes conducted in an atmosphere of impure hydrogen containing hydrocarbons, moisture and oxygen, the surface of the steel beneath the insulating magnesium oxide layer is attacked forming inclusions of interstitial carbides and oxides thereby impairing the magnetic properties of the steel. This quite obviously is a serious problem which demands a particular type of insulation to preserve the necessary magnetic properties.

A further problem arises because magnesium oxide is applied to steel from an aqueous suspension or slurry. During the initial stages of the box annealing process, water volatilizes from an applied coating and can under ordinary conditions react with the iron on the surface of the steel forming current conducting iron oxides which impair the electrical insulating properties sought. Areas on the surface of the steel displaying such deleterious iron oxide formations are referred to in the art as "anneal patterns." Furthermore, it has been found that magnesium oxide does not form an intimately bonded tightly adherent coating which is not easily removed upon contact.

It has been found that magnesium oxide per se fails to provide the desired high degree of required insulation and magnetic protection properties to overcome the noted defects. Accordingly, it would be desirable to provide magnesium oxide compositions for coating steel which compositions are tightly adherent and maintain suitable electrical insulating properties after box annealing and stress relief annealing, and which also preserve the desired magnetic properties of the steel after stress relief annealing.

It is therefore an object of the present invention to provide improved magnesium oxide compositions for coating silicon steel which compositions form electrical insulating barriers that prevent the formation of undesirable anneal patterns believed to be due to iron oxide formations during box annealing.

It is another object of the present invention to provide magnesium oxide compositions for coating silicon steel which compositions form adherent electrical insulating coatings which are also capable of preventing attack of the steel surface by impure hydrogen during stress relief annealing.

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It is still another object of the present invention to provide magnesium oxide coating compositions for coating silicon steel which compositions have the above attributes and also function as efficient separator media for coiled, rolled steel sheet.

It is a further object to provide a simplified process for coating steel sheet with said magnesium oxide compositions to achieve the aforementioned benefits.

The fulfillment of these and other related objects of this invention may be more readily appreciated by refer- 10 ence to the following specification, examples, and appended claims.

SUMMARY OF THE INVENTION

Broadly, the above objects are accomplished by the 15 provision of magnesium oxide coating compositions containing a major proportion of magnesium oxide, and a minor proportion of the additives:

- (I) a boron containing compound selected from the group consisting of boric acids, boric oxides, and magnesium 20 The molar ratio of magnesium oxide to silica is from borates;
- (II) a sodium compound selected from the group consisting of sodium hydroxide, sodium oxide, sodium peroxide and sodium borate; and
- (III) a silicon-containing compound selected from the 25 group consisting of silica and alkaline earth metal silicates.

The invention also provides a process for coating steel sheet with an adherent, electrical insulating, corrosion-30 resistant coating comprising in sequence applying to the surface of said steel an aqueous slurry containing the said magnesium oxide coating composition, heating to remove water therefrom, and thereafter annealing the coated steel at a temperature in excess of about 1000° C.

35The magnesium oxide employed has a surface area in excess of about 20 square meters per gram or an iodine number (iodine adsorption valve) in excess of about 24 milligrams of iodine per gram of dry magnesium oxide. The surface area of magnesium oxide as referred to herein $_{40}$ is determined by the standard Brunaeur-Emmet-Teller method. The iodine number is approximately 1 to 1.25 times the surface area in square meters per gram.

The iodine number as referred to herein is determined by the following procedure:

METHOD OF IODINE NUMBER DETERMINATION

- (1) Weight a 2 gram sample of magnesium oxide to the nearest milligram.
- (2) Transfer to a clear, dry, 200-ml. glass-stoppered bottle.
- (3) Add 100±0.2 ml. of 0.100 N iodine in carbon tetrachloride, free from traces of sulfur or carbon disulfide.
- (4) Stopper the bottle and shake vigorously at ambient temperature in a suitable shaking device for 30 minutes (the test is relatively insensitive to temperature so that 55no temperature controls are employed).
- (5) Allow to settle for 5 minutes or longer and then pipette a 20 ml. aliquot of the clear solution into a 250 ml. Erlenmeyer flask containing 50 ml. of 0.03 N potassium iodide in 75 percent ethanol.
- (6) Titrate the 20 ml. aliquot with standard 0.05 N sodium thiosulfate. The sodium thiosulfate should be standardized at least once every two weeks against a standard potassium iodate solution. A sharp end point is obtained without the use of starch indicator.
- (7) Calculate iodine number in terms of milligrams of iodine per gram of sample according to the following equation:

$318(V_2 - V_1)N_1 = MgI_2/g = iodine number$

where V_2 is the volume of thiosulfate equivalent to 20 ml. of the original iodine solution-before adsorption of iodine by the oxide; where V_1 is the volume of thiosulfate required by the 20 ml. aliquot after adsorption; and N_1 is the normality of the thiosulfate solution.

By the term "boric acids" it is meant to include all of the various known boric acids, such as for example, but not limited to, metaboric acid, orthoboric acid and pyroboric acids.

By the term "magnesium borates" it is meant to include the various known magnesium borates, such as for example, but not limited to the magnesium orthoborates, the magnesium metaborates and the magnesium pyroborates.

By the term "sodium borate" it is meant to include all of the various known sodium borates, such as for example, but not limited to sodium tetraborate, sodium metaborate, and sodium orthoborate. Silica in virtually any form and having a particle less than about 200 mesh may be employed, such as the product available under the trade name "Cabosil." Equally operable are the alkaline earth metal silicates, such as for example, calcium silicate and magnesium silicate.

Generally, the concentrations of additives, on a molar ratio basis, range as follows:

- about 280:1 to about 2900:1, preferably from about 1000:1 to 2000:1;
- The molar ratio of boron (expressed as B₂O₃) to silica is from about 3:1 to about 5:1, preferably from about 3:1 to 4:1:
- The molar ratio of magnesium oxide to sodium is from about 2000:1 to about 4000:1 with about 2800:1 being preferred.

In the aggregate the additives (silica, boron and sodium, calculated as expressed above) in the coating composition may range up to about 21/2 mole percent and are preferably present in amounts up to about 2 mole percent, based on the total moles of MgO and additives.

For coating steel, an aqueous suspension of the above described magnesium oxide composition is prepared by mixing with water to the desired viscosity and leveling and flow-out characteristics. Generally from about 5 to about 20 weight percent of the magnesium oxide composition, based on water, is satisfactory to provide an aqueous slurry having the requisite viscosity and flow properties suitable for coating onto steel sheet.

A frit may also be prepared by fusing magnesium oxide with a boron-containing compound, a sodium compound and a silicon-containing compound as hereinbefore described. The frit may then be comminuted to a particle size less than 200 mesh and added to water to form the coating slurry. In preparing aqueous slurries of the coating compositions, the order of addition or admixture of the various components of the composition is immaterial.

The coating slurry may be applied to the magnetic sheet material by any suitable means such as by immersion, brushing, or spraying. It has been found convenient to use an immersion technique whereby the steel sheet is passed through a tank containing the coating slurry. The coated sheet thereafter is heated to drive off water and provide a dried layer of the present composition. The coated metal sheet is then coiled into a roll and placed in a furnace for box annealing as previously described. During the annealing process, the coating of this invention forms an ad-60 herent, electrically insulating, corrosion-resistant layer which also functions as a separator medium to prevent the coiled metal sheet from sticking to itself.

BRIEF DESCRIPTION OF THE DRAWING

65 The drawing is a schematic diagram of a conventional process for coating the compositions of the present invention on steel strip. As therein illustrated, a magnesium oxide composition contained in hopper 10 and water in tank 12 are fed via 13 and 13a to mixing tank 14 to form 70 an aqueous coating suspension or slurry. The aqueous

coating slurry is conducted through line 18 equipped with valve 20 to coating trough 22. Newly formed, hot steel sheet 23 is passed in the direction indicated by arrows over roller 24 and under roller 26 through quench tank 75 28 and thence over roller 25 and through coating trough 10

22 where it acquires a layer of the coating slurry. The steel sheet coated with an aqueous slurry of the coating composition is then passed between adjustable pinch rollers 30 which regulate the coating thickness and then to heater 32. In the heater, water is volatilized from the slurry layer on the steel strip to provide an adherent coating. The coated steel sheet is passed over roller 33 and coiled into suitable lengths at take-up roll 34. The coated coiled steel roll 23A is thereafter passed to annealing furnace 36, and thence to storage.

DESCRIPTION OF THE PREFERRED **EMBODIMENTS**

In one preferred form, a steel coating composition comprising magnesium oxide, boric acid, sodium hydroxide, 15and silica is provided in which the molar ratio of magnesium oxide to silica is about 1500:1, the molar ratio of boric oxide to silica is about 3.5:1 and the molar ratio of magnesium oxide to sodium is about 2800:1. Approximately 10 to 12 weight percent of this composition is ad- 20 mixed with water to form a slurry suitable for coating onto steel sheet. Coiled steel sheet which was coated with this slurry and subsequently dried and thereafter box annealed according to the procedure hereinbefore described was easily unrolled. Further, the surface of the steel was 25 relatively electrically non-conducting and resistant to attack by corrosive gases or fumes.

In another preferred form a magnesium oxide composition having the aforenoted desirable steel coating properties is provided which comprises magnesium oxide, boric 30 oxide, sodium tetraborate, and silica in which the molar ratio of MgO to silica is about 1500:1, the molar ratio of boric oxide to silica is about 3.5:1, and the molar ratio of magnesium oxide to sodium is about 2800:1.

For a more complete understanding of the present in- 35 vention, reference is now made to the following specific examples illustrating the improved properties of the coating compositions of the present invention.

EXAMPLE 1

A steel coating suspension was prepared by adding 18.0 grams of MgO (0.45 mole) to 150 ml. of an aqueous boric acid solution containing 0.00105 mole of boron expressed as B_2O_3 . To this suspension was added 0.00016 mole of sodium hydroxide and 0.0003 mole of silica having a crystallite size of about 70 A., to yield an aqueous coating composition of about 10% solids containing the following components in the indicated ratios:

| Coating Composition A: | Molar ratio | 50 |
|------------------------|-------------|----|
| MgO:SiO ₂ | 1500:1 | |
| $B_2O_3:SiO_2$ | 3.5:1 | |
| MgO:Na | 2812:1 | |

Coating procedure

The resultant suspension was subjected to constant stirring and was coated onto 26 separate silicon steel sheets or plates, each sheet measuring 3.0 x 15.3 cm. by 12-14 mils in thickness. The coatings were leveled and 60 then dried in air at a temperature of about 500° C. The coated, dried sheets were then stacked one upon another and annealed by heating in a hydrogen atmosphere at a temperature of 1177° C. for a period of 4.3 hours. The annealed sheets were then cooled, and excess or loose 65 magnesium oxide was scrubbed from the surface of each coated sheet by brushing in a stream of flowing water.

The procedure was repeated except that the ratio of MgO:SiO₂ in the coating composition was increased to 2960:1 as indicated:

| Coating Compos | ition B: 1 | Molar | ratio |
|----------------------|------------|-------|-------|
| MgO:SiO ₂ | | 29 | 960: |
| $B_2O_3:SiO_2$ | | | 3.5:1 |

MgO:Na _____ 2800:1 75

The conductance of the coated surfaces of the sheets coated with Coating Composition A and Coating Composition B respectively was measured by means of the Franklin test.

Franklin test

This test is widely accepted and utilized for evaluating the conductance of coated steel sheets. A detailed description is found in ASTM method A-334-52, "Standard Methods of Test for Electrical and Mechanical Properties of Magnetic Materials." Briefly, the test is carried out by passing an electric current through brass contacts which cover coated areas 0.1 square inch in area. Current passing through the coating flows through the steel to a contact made directly to the steel by means of a twist drill. The resulting amperage provides a measure of the resistance encountered through the coating. Several hundred contacts are employed in obtaining readings for coating evaluation. A complete short circuit is indicated by a reading of 100 milliamps per 0.1 square inch.

For the coatings of the present invention, Franklin test values of about 45 or less are considered acceptable. The results obtained for Coating Compositions A and B respectively are as follows:

| | Franklin test |
|-----------------------|---------------|
| | milliamps/ |
| | 0.1 sq. in. |
| Coating Composition A | 26 |
| Coating Composition B | 52 |
| | |

EXAMPLE 2

The procedure of Example 1 was repeated except that the coating composition contained the following components in the indicated ratios:

| Coating Compo | sition C: | Molar | ratio |
|----------------------|-----------|-------|-------|
| MgO:SiO ₂ | | 14 | 480:1 |
| $B_2O_3:SiO_2$ | | | 4.6:1 |
| MgO:Na | | 28 | 830:1 |

The procedure of Example 1 was repeated except that the coating composition contained the following components in the indicated ratios:

| Coating Compo | osition D: | Molar | ratio |
|----------------------|------------|-------|-------|
| MgO:SiO ₂ | | 1. | 480:1 |
| $B_2O_3:SiO_2$ | | | 8.7:1 |
| MgO:Na | | 2 | 830:1 |

The coated annealed steel plates were subjected to the Franklin Test with the following results:

| | Franklin to | est |
|-----------------------|-------------|-----|
| | milliamp | os/ |
| | 0.1 sq. | in. |
| Coating Composition C | | 26 |
| Coating Composition D | | 58 |
| | | |

EXAMPLE 3

The procedure of Example 1 was repeated except that the ratio of B_2O_3 to SiO₂ employed was 4.6:1 to provide a coating composition having the following components in the indicated ratios:

| Coating Compo | sition E: | Molar ratio |
|----------------|-----------|-------------|
| $MgO:SiO_2$ | | 1480:1 |
| $B_2O_3:SiO_2$ | | 4.6:1 |
| MgO:Na | | 2800:1 |

The coated steel plates were observed for uniformity of coating by visual inspection.

The procedure of Example 1 was repeated except that the ratio of B₂O₃ to SiO₂ was 2.3:1 to provide a coating composition having the following components in the in-70 dicated ratios:

| Coating Compo | osition F: M | olar ratio |
|----------------------|--------------|------------|
| MgO:SiO ₂ | | _ 1480:1 |
| $B_2O_3:SiO_2$ | | _ 2.3:1 |
| MgO:Na | | _ 2800:1 |

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The coated steel plates were observed for uniformity of coating by visual inspection. The results were as follows:

| | Molar ratio (B ₂ O ₃ -SiT ₂) | Uniformity of coating | 5 |
|-----------------------|-------------------------------------------------------------------|-----------------------|---|
| Coating Composition E | 4.6:1 | Excellent. | Ŭ |
| Coating Composition F | 2.3:1 | Bare spots. | |

EXAMPLE 4

The procedure of Example 1 was repeated except that 10 the ratio of MgO to sodium was varied while keeping the ratios of MgO:SiO₂ and B₂O₃:SiO₂ constant to provide coating compositions having the following components in the indicated ratios:

| Coating Composition G: | Molar ratio | 15 |
|-------------------------------------------------|-------------|----|
| MgO:SiO ₂ | 1480:1 | |
| B ₂ O ₃ :SiO ₂ | 3.5:1 | |
| MgÖ:Na | 1415:1 | |
| Coating Composition H: | Molar ratio | 20 |
| MgO:SiO ₂ | 1480:1 | |
| B ₂ O ₃ :SiO ₂ | 3.5:1 | |
| MgÖ:Na | 5660:1 | |
| | | |

Steel plates were coated with Coating Compositions A, G and H respectively, and were examined visually for 25 uniformity of coating with the following results:

| | | | Joanne | |
|------------|-------------|---|-----------|----|
| Coating Co | mposition A | A | Good. | |
| Coating Co | mposition (| G | Poor. | 30 |
| Coating Co | mposition I | H | Poor. | |
| | | | | |

Tiniformity of conting

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EXAMPLE 5

Coa

A coating composition was prepared as in Example 1, except that the molar ratio of MgO:SiO₂ was as indicated: 35

| ting Compos | ition I: M | olar ratio |
|-------------------------------------------------|------------|------------|
| MgO:SiÔ ₂ | | _ 2830:1 |
| B ₂ O ₂ :SiO ₂ | | _ 3.5:1 |
| MaO·Na | | _ 2800:1 |

The procedure of Example 1 was followed in coating this composition onto steel plates. A control consisting of MgO with no additives was also coated onto steel plates according to the same procedure. The coated steel plates were then tested for corrosion resistance by means of the Chlorine Test for Corrosion Resistance.

Chlorine test for corrosion resistance

In this test strips of weighed steel having 30 cm.² of surface area rest against a thermocouple well located at the center of the hot zone of a heated Vycor tube in an 50 automatically controlled tube furnace. Helium flows through the tube at a linear velocity of 65 cm./min. The temperature is raised to 816° C. to simulate stress relief annealing conditions and 10 minutes are allowed for equilibrium to be established after the temperature levels 55 off. The gas is then suddenly switched to wet chlorine moving at 15 cm./min. After 60 seconds, the flow is switched back to a 170 cm./min. flow of helium. The system is then cooled to 315° C., samples removed, quenched in cold running water, scrubbed, dried and reweighed. 60 The weight loss due to iron and silicon chloride vaporization is an inverse measure of the corrosion resistance.

The results obtained were as follows:

| | Weight loss, grams | Reduction in weight loss, compared to control | Percent improve- ment | 65 |
|-----------------------|--------------------------|--------------------------------------------------------|-----------------------------|----|
| Coating Composition I | 0.18 0.32 | 0.14 | 43.8 | 70 |

EXAMPLE 6

A coating composition was prepared as in Example 1 except that sodium tetraborate was used instead of sodium hydroxide and the molar ratio of MgO:SiO2 was 75 tion M) was prepared according to the procedure of

943:1 to yield a composition having the following components in the indicated ratios:

| Coating Compo | sition J: | Molar ratio |
|-------------------------------------------------|-----------|-------------|
| MgO:SiO ₂ | | 943:0 |
| B ₂ O ₂ :SiO ₂ | | 3.5:1 |
| MgO:Na | | 2800:1 |

The procedure of Example 1 was followed in coating this composition onto steel plates except that the coated plates were annealed for a period of 8.6 hours. The coated, annealed plates were tested for corrosion resistance according to the procedure described in Example 5. The results obtained were as follows:

| Weight loss, grams | Reduction in weight loss, compared to control | Percent improve- ment |
|--------------------------|--------------------------------------------------------|------------------------------------------------------------|
| 0.21 0.32 | 0.11 | 34.4 |
| | Weight loss, grams 0.21 0.32 | Reduction in weight loss, grams control 0.21 0.11 |

EXAMPLE 7

A coating composition was prepared as in Example 1 (hereinafter coating composition \bar{K}) except that sodium tetraborate was used instead of sodium hydroxide and the molar ratio of MgO:SiO₂ was 2830:1 to yield a composition having the same ratios of components as the composition of Example 5.

The procedure of Example 1 was followed in coating this composition onto steel plates except that the coated plates were annealed for a period of 8.6 hours. The coated, annealed plates were tested for corrosion resistance according to the procedure described in Example 5.

The results obtained were as follows:

| | Weight loss, grams | Reduction in weight loss, compared to control | Percent improve- ment |
|----------------------------------|--------------------------|--------------------------------------------------------|-----------------------------|
| Coating Composition K Control | 0, 17 0, 32 | 0.15 | 46.9 |

EXAMPLE 8

A composition (hereinafter coating composition L) was prepared by fusing together magnesium metaborate, $Mg(BO_2)_2$, and SiO₂ to prepare a frit. The frit was then comminuted and suspended in an aqueous solution containing sodium hydroxide and boric acid in such proportions to yield a composition having the same ratios of components as coating compositions I and K.

The procedure of Example 1 was followed in coating this composition onto steel plates except that the coated plates were annealed for a period of 8.6 hours. The coated, annealed plates were tested for corrosion resistance according to the procedure described in Example 5.

The results obtained were as follows:

| 5 | Weight loss, grams | Reduction in weight loss, compared to control | Percent improve- ment |
|-----------------------|--------------------------|--------------------------------------------------------|-----------------------------|
| Coating Composition L | 0.22 0.32 | 0.10 | 31, 2 |

EXAMPLE 9

A coating composition (hereinafter coating composi-

Example 1 except that the molar ratio of MgO:SiO₂ was 283:1 to yield a composition having the following components in the indicated ratios:

| Coating Composition M: M | Iolar ratio |
|--------------------------|-------------|
| MgO:SiO ₂ | _ 283:1 |
| $B_2O_3:SiO_2$ | _ 3.5:1 |
| MgO:Na | _ 2800:1 |

The procedure of Example 1 was followed in coating this composition onto steel plates except that the coated 10 molar ratio of magnesium oxide to silica is from about plates were annealed for a period of 8.6 hours. The coated, annealed plates were tested for corrosion resistance according to the procedure described in Example 5.

The results obtained were as follows:

| | Weight loss, grams | Reduction in weight loss compared to control | Percent improve- ment | 15 |
|----------------------------------|--------------------------|-------------------------------------------------------|-----------------------------|----|
| Coating Composition M Control | 0, 19 0, 32 | 0, 13 | 40.9 | 20 |

EXAMPLE 10

A steel coating composition (hereinafter coating composition N) was prepared by admixing magnesium oxide, boric oxide, sodium tetraborate and silica in the follow- 25 ing molar ratios: Malan notic

| | lar ratio | N: Mo | osition | Comp | Coating |
|----|-----------|-------|---------|--------------------|---------|
| | 1480:1 | | | O:SiO ₂ | Mg |
| 30 | 3.5:1 | | | 3:SiO2 | B_2C |
| 00 | 2800:1 | | | O:Na | Mg |

An aqueous slurry of Composition N was prepared and the procedure of Example 1 was followed in coating this composition onto steel plates and thereafter annealing the coated plates. The coated, annealed plates were tested 35 insulating, corrosion-resistant coating which comprises in for corrosion resistance according to the procedure described in Example 5.

The results obtained were as follows:

| | Weight loss, grams | Reduction in weight loss compared to control | Percent improve- ment | 4(|
|----------------------------------|--------------------------|-------------------------------------------------------|-----------------------------|----|
| Coating Composition N Control | 0.09 0.32 | 0. 23 | 72 | 4 |

While several particular embodiments of this invention are shown above, it will be understood, of course, that the invention is not to be limited thereto, since many modifications may be made, and it is contemplated, there-50 fore, by the appended claims, to cover any such modifications as fall within the true spirit and scope of this invention.

What is claimed is:

1. A magnesium oxide composition containing a ma- 55 jor proportion of magnesium oxide having a surface area of at least 20 square meters per gram and a minor proportion of the additives:

- (I) a boron compound selected from the group consisting of boric acids, boric oxides, and magnesium 60 borates:
- (II) a sodium compound selected from the group consisting of sodium hydroxide, sodium oxide, sodium peroxide and sodium borate; and
- (III) a silicon-containing compound selected from the 65 group consisting of silica and alkaline earth metal silicates:

wherein the molar ratio of magnesium oxide to silica is from about 280:1 to about 2900:1, the molar ratio of boron expressed as (B_2O_3) to silica is from about 3:1 to about 5:1, and the molar ratio of magnesium oxide to sodium is from about 2000:1 to about 4000:1.

2. A composition according to claim 1 wherein the boron compound is boric acid, the sodium compound is

sodium hydroxide, and the silicon-containing compound is silica.

3. A composition according to claim 1 wherein the molar ratio of magnesium oxide to silica is from about 1000:1 to about 2000:1, the molar ratio of boron expressed as B₂O₃ to silica is from about 3:1 to about 5:1, and the ratio of magnesium oxide to sodium is about 2000:1 to 4000:1.

4. A composition according to claim 1 wherein the 1000:1 to about 2000:1, the molar ratio of boron expressed as B₂O₃ to silica is from about 3:1 to about 4:1, and the ratio of magnesium oxide to sodium is about 2000:1 to about 4000:1.

5. A composition according to claim 4 wherein the molar ratio of magnesium to sodium is about 2000:1.

6. A composition according to claim 1 wherein the aggregate mole percentage of the additives boron, silica and sodium to the total composition is up to about $2\frac{1}{2}$ o mole percent.

7. A composition according to claim 1 wherein the aggregate mole percentage of the additives boron, silica and sodium to the total composition is up to about 2 mole percent.

8. A composition according to claim 1 wherein the molar ratio of magnesium oxide to silica is about 1500:1, the molar ratio of boric acid expressed as B₂O₃ to silica is about 3.5:1, and the molar ratio of magnesium oxide to sodium is about 2800:1.

9. A composition according to claim 1 wherein the boron compound is boric acid, the sodium compound is sodium tetraborate, and the silicon-containing compound is silica.

10. A process for coating steel sheet with an electrically sequence forming an aqueous slurry of a magnesium oxide composition as defined in claim 1, coating said slurry to the surfaces of steel sheet, heating the slurry coated steel to dry said coating, and thereafter annealing 0 said coated steel sheet.

11. A process according to claim 10 wherein the slurry has a solids content of from 5 to 20 percent by weight.

12. A process according to claim 10 wherein the silica has a particle size of up to 200 mesh (U.S. Standards 5 Sieve Series).

13. A process according to claim 10 wherein the steel sheet is coiled after coating and before the annealing step.

14. A process according to claim 10 wherein the boron compound is boric acid, the silicon-containing compound is silica, and the sodium compound is sodium hydroxide.

15. A coated steel sheet having thereon an adhered coating of the composition defined in claim 1.

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