This invention relates to coated material, a method for applying an adherent coating to the surface of strip material, and in particular to the method for applying protective coating to the surface of magnetic material.

In the past there have been numerous attempts made to improve the protective coatings applied to strip materials during various heat treatment processes. In particular, magnetic materials have been treated in order to provide the required degree of insulation as well as protecting the material when it is placed in contact with itself, for example, when the material is cooled preparatory to high temperature box annealing. The most prevalent coatings employed on magnetic materials have consisted of slurries formed from hydrated MgO. Research has centered about the aspects of the character of the MgO, its particular size, calcining temperature, the method of its application and the rate of its application, said rate being particularly concerned with the amount of hydration to which the MgO has been subjected, as well as the amount of water carried over into the final high temperature heat treatment. The property sought in providing coatings to magnetic materials has been the insulation characteristic without detrimentally affecting the magnetic characteristics. Moreover, these coatings have value as a separator during the formation of the insulating coating. The most difficult aspect with all of the prior art coatings has resolved itself about the aspect of providing sufficient adhesion to the strip material during its processing intermediate the application of the coating as a wet slurry and prior to the reaction thereof with the magnetic material to form the insulating coating. Particular care has heretofore been required in order that the coating as applied to the magnetic material did not come in contact with any equipment prior to its heat treatment, because of the possibility of disturbing the uniform thickness of the coating and of wiping the coating from the strip material.

An object of the present invention is to provide a strip material with an improved coating which is tightly adherent and can be subjected to normal handling operations prior to heat treatment.

Another object of the present invention concerns the provision of magnetic material with an improved insulating coating.

A further object of the present invention is to provide magnetic material having an improved MgO coating thereon which, in its unfired condition, can be subjected to normal mill handling without adversely affecting its final characteristics.

A more specific object of the present invention is to provide a hydrated MgO coating to the surface of silicon steel which can react to provide superior insulation, which insulating coating is more uniform over the cross section of the strip material.

In its broader aspects, this invention contemplates the provision for applying a slurry coating to the basic metal in order to form a protective coating thereon. The coating, as thus applied, is metered to obtain a uniform thickness and thereafter it is dried with gentle heat. With the excess water removed, the metal having the "green" coating thereon is subjected to a roll compacting operation following which the metal is thereafter subjected to a heat treatment.

More specifically, the invention is particularly directed to the production of magnetic material having an improved insulating coating thereon. These magnetic materials include the silicon steels, nickel-irons, and various of the other magnetic materials. The invention is also useful in retarding oxidation in stainless steel and other exotic metals which are heated to elevated temperatures. In the past, MgO of various characteristics and various states of hydration has been employed as the insulating coating on silicon steel. The MgO apparently reacts with silica which is formed on the surface of silicon steels, and during the heat treatment magnesium silicate is formed which acts as an insulating glass. This insulating glass is tightly adherent to the surface of the silicon steel. The manner of the application of the MgO may be varied from an immersion or contact-type application to that of charging the MgO in an electrostatic field or using an ionized form of MgO as an electrolyte and passing the material through an electrolytic cell. For the purposes of the present invention, it is immaterial how the MgO is placed upon the surface of the material so long as said coating is uniform. Obvious economies are effected where the process is continuous, for example, the coating of strip material.

In particular, excellent results have been obtained where the hydrated MgO is placed on the surfaces of strip material by immersing the strip in a container wherein the slurry is maintained. Upon withdrawal from said container, the strip may be passed through metering rolls which will control the uniformity and thickness of the slurry so that the MgO is preferably present in an amount ranging between about 0.025 and about 0.035 oz. per square foot of strip material. While these values are not critical, nonetheless they appear to be preferable, and good results have been obtained by employing such concentrations of MgO.

Where desired, the slurry which is applied to the strip material may also contain special purpose additives, for example, formic acid and chromic acid. The additives may vary the rate of hydration of the MgO, or they may foster slightly better adhesion of the "green" coating or prevent the oxidation of the strip material during the application of the slurry thereto. The additives may also perform various other functions so long as they do not interfere with or detract from the basic insulating characteristics which are possible from the reaction product of MgO with the silica contained on the surface of silicon steel magnetic strip material. Additives may also perform different functions depending upon the base material to which the slurry coating is applied, for example, in the case of stainless steels or nickel-irons, the additives may give increased adhesion of the "green" coating to the surface of the material to which the slurry is applied. In addition, various other additives may provide some measure of further protection which will cooperate with the alkali earth metal oxide as employed in the process of the present invention to obtain a measure of insulation to the material to which the coating is applied.

Following the application of the slurry to the surface of the material and the metering thereof to obtain a substantially uniform coating of the alkali earth metal oxide, it is preferred to subject the material to a drying furnace preferably operating at a temperature of up to about
1600° F. maximum. While preferably the drying furnace operates at a temperature of approximately 1200° F., the temperature of the material will usually be at between 500 and 700° F. and preferably at about 600° F., and this is accomplished by passing the material continuously through said furnace and at a rate so that the material is within the hot zone of the furnace for a relatively short period of time, i.e., for a period ranging between about 12 hours and about 72 hours. During the box anneal a dry hydrogen atmosphere is employed, said atmosphere having a dew point of less than about −70° F. At the completion of the box annealing cycle, the material may be cooled to room temperature from where it may be processed further as desired by the ultimate user of the magnetic material.

The foregoing has been described in particular with respect to magnetic material which has been decarburized and given a final normalizing heat treatment prior to coating as described aforesaid. In this particular respect, care must be exercised during the roll compacting operation so that the stress is limited to such a degree that no untoward stress is applied to the material which would adversely affect its magnetic characteristics. An example of the processing as set forth hereinafter was employed on 3-inch wide material which was compacted using 10° diameter rolls. The following results were obtained:

<table>
<thead>
<tr>
<th>TABLE I</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td>Gauge as coated</td>
</tr>
<tr>
<td>Gauge as compacted</td>
</tr>
<tr>
<td>Total Mill Force</td>
</tr>
<tr>
<td>Franklin (tonm. at 0.440° F.)</td>
</tr>
<tr>
<td>Core loss at 15 KHz</td>
</tr>
<tr>
<td>At 60 Hz</td>
</tr>
<tr>
<td>At 40 Hz</td>
</tr>
</tbody>
</table>

From the test results recorded in Table I, it is clear that while slight change in gauge is noted, superior insulating properties are obtained, improved core loss is apparent, and the permeability is altered as indicated. Additional disk tests indicate that the basic steel has the same relative degree of orientation in both the uncoated and coated conditions.

As a preferred alternate embodiment of the method of the present invention, it is preferred to employ finish gauge cold rolled material and employ the process of the present invention before subjecting the material to a final heat treatment. The following schedule lists the method of the present invention as applied to cold rolled strip material of finish gauge.

1. Degreasing and/or pickling.
2. Coat with slurry formed of about 9% solid MgO, balance water. Alternately, up to 60 ml. of formic acid per gallon and 13 grams of CrO4 per gallon may be added.
3. Meter slurry on at least one or both surfaces of the strip material so that the concentration of MgO on the strip material is within the range between 0.025 and 0.035 oz./ft².
4. Pass strip through drying furnace operating at about 1200° F., said strip being in hot zone and not over one minute.
5. Roll compact the dried “green” coating employing a force between 500 and 8,000 pounds per inch of strip width.
6. Normalize strip material at 1475° F. for a time period of up to 3 minutes.
7. Box anneal at a temperature of 2150° F. for a time period ranging between 12 and 72 hours and employing a hydrogen atmosphere having a dew point of less than about −70° F.

The material as processed on a continuous basis and employing cold rolled material of finish gauge can be roll compacted to a greater degree without affecting the magnetic characteristics of the strip material. Accordingly, the roll compacted strip, prior to the normalize and box anneal, has a glossy appearance, feels waxy to the touch, and has great adhesion, as determined by the “bend and blow” test.

The following Table II lists the comparative properties of cold rolled strip material which has been coated using the process of the present invention in comparison with
Section A of Table II gives the results of the Franklin values of the material processed both according to the prior art practice and by employing the process of the present invention. It is to be noted that ten samples were employed in which the Franklin values were recorded on both the top and bottom of the individual samples and, as set forth therein, the average value employing the prior art practice was about .77. Contrasted thereto, when the process of the present invention was employed on the same number of samples which were tested for their Franklin values, it became quite clear that the average value was .35. Since these tests were conducted in accordance with A.S.T.M. designation A-344-60T wherein a reading of 0 indicates perfect insulation, and a reading of 1 is a complete short circuit, it is clear that the process of the present invention is effective for producing an outstanding degree of insulation in the coating when the process as set forth hereinbefore is employed for coating the strip materials. Referring now to sub-section B of Table II, representative values of the magnetic characteristics are included which clearly demonstrate that the process of the present invention does not adversely affect the magnetic characteristics, and in some instances clearly improves the magnetic characteristics exhibited when the process of the present invention is employed in comparison with prior art practices. While there were substantially insignificant differences in the permeability as measured at 10H, some improvement was noted in the core loss, as well as the permeability, when measured at 200H. Thus, the process of the present invention is effective for providing an insulating coating to magnetic strip material without adversely affecting the magnetic characteristics.

From the physical characteristics of the material processed according to the method of the present invention, as well as by the "bend and blow" test, the present process is effective for greatly increasing the adherence of the protective coating to the base material to which it is applied. This will facilitate normal handling of the coated strip in steel mill operations without damage to the coating. Moreover, the process as applied to decarburized cold rolled silicon steel is also effective for decreasing the total water content of the coating with the result that lower amounts of moisture are evolved during the high temperature box annealing heat treatment, thereby resulting in better magnetic characteristics being developed in these magnetic materials. In addition, roll compacting minimizes variations in the quality of the alkali earth metal oxide powder which is employed in the protective coating. These differences in quality are reflected, not only in the powder size and rate of hydration, but also in the character of the insulating characteristics finally developed on the magnetic strip material. As will be appreciated, the method of the present invention is not limited to magnetic materials but the same may be extended to exotic magnetic materials as an oxidation retardant. This results from the fact that even after roll compacting, unless there is a reaction between the base metal and the MgO of the coating after high temperature annealing as in the case of silicon steel, the coating becomes quite friable and easily stripped from the base material. In this respect, the roll compacting provides a much denser coating during heat treatment, thereby improving the oxidation resistance of the product.

I claim:

1. In the method of forming a protective coating on silicon steel strip material, the steps comprising, in sequence, applying a uniform coating containing hydrated magnesium hydroxide, 0.025 and 0.035 oz. per square foot of strip surface to the surface of the strip, roll compacting the coated strip, and heat fixing the coating on the strip.

2. In the method of forming an insulating coating on silicon steel strip material, the steps comprising, in sequence, applying a uniform coating containing hydrated magnesium hydroxide, 0.025 and 0.035 oz. per square foot of strip surface, drying the strip at a temperature in the range between about 500° F. and about 700° F., roll compacting the coated strip, and heat fixing the coating on the strip.

3. In the method of forming an insulating coating on magnetic steel strip material, the steps comprising, in sequence, applying a uniform coating containing hydrated magnesium hydroxide and formic acid to the surface of the magnetic strip material in a concentration within the range between 0.025 and 0.035 oz. of magnesium hydroxide per square foot of strip surface, passing the uniformly coated magnetic strip material through a drying furnace operating at a temperature within the range between about 500° F. and about 700° F., roll compacting the dried magnetic strip material by passing the same through a conventional rolling mill while exerting a pressure ranging between about 500 and 3000 pounds per inch of strip width, normalizing the roll compacted magnetic strip material, and thereafter box annealing the normalized steel strip.

4. In the method of forming an insulating coating on silicon steel strip, the steps comprising, in sequence, applying a uniform coating containing hydrated magnesium hydroxide and a rust inhibitor to the surface of the silicon steel strip in a concentration within the range between 0.025 and 0.035 oz. of magnesium hydroxide per square foot of strip surface, passing the uniformly coated strip through a drying furnace to heat the strip to a temperature within the range between about 500° F. and about 1475° F. for a time period ranging between about 1 minute and about 4 minutes, and thereafter box annealing the normalized steel strip within the range between about 2150° F. and about 2300° F. for a time period of from about 12 to about 72 hours.

5. In the method of forming an insulating coating on silicon steel strip, the steps comprising, in sequence, applying a uniform coating containing hydrated magnesium hydroxide, 0.025 and 0.035 oz. per square foot of strip surface, drying the strip at a temperature in the range between about 500° F. and about 700° F., roll compacting the coated strip, and heat fixing the coating on the strip.
hydroxide to the surface of the silicon steep strip in a concentration within the range between 0.025 and 0.035 oz. of magnesium hydroxide per square foot of strip surface, passing the uniformly coated strip through a drying furnace operating at a temperature within the range between about 800° F. and about 1600° F., roll compacting the dried strip by passing the same through a conventional rolling mill while exerting a pressure ranging between about 500 and 3000 pounds per inch of strip width, normalizing the rolled compacted silicon steel strip in a furnace operating at a temperature within the range between about 1200° F. and about 1475° F. for a time period ranging between about 1 minute and about 4 minutes, and thereafter box annealing the normalized steel strip at a temperature within the range between about 2150° F. and about 2300° F. for a time period of from about 24 to about 60 hours.

6. In the method of forming a protective coating on metallic strip material, the steps comprising, in sequence, applying a uniform coating containing alkali earth metal hydroxide to the surface of the metallic strip material, roll compacting the coated strip, and thereafter heat fixing the coating on the strip.

7. In the method of forming a protective coating on silicon steel strip material, the steps comprising, in sequence, applying a slurry formed from water containing from about 7% to 12%, by weight of MgO, from about 10 to about 16 grams of CrO₃ per gallon of water, and from about 50 to 80 ml. of formic acid per gallon of water to the surface of the strip, drying the strip, roll compacting the slurry coated strip, normalizing the steel strip, and box annealing the steel strip.

8. In the method of forming a protective coating on metallic material the steps comprising, in sequence, applying a uniform coating containing alkali earth metal hydroxide to the surface of the metal in a concentration within the range between 0.025 and 0.035 oz. per square foot of surface, passing the uniformly coated material through a drying furnace and roll compacting the dried material by passing the same through a conventional rolling mill while exerting a pressure ranging between about 500 and 3000 pounds per inch of strip width.

9. In the method of forming an insulating coating on silicon steel strip material, the steps comprising, in sequence, applying a uniform coating containing magnesium hydroxide to the surface of the finished gauge cold rolled silicon steel strip in a concentration within the range between 0.025 and 0.035 oz. of magnesium hydroxide per square foot of strip surface, passing the uniformly coated strip through a drying furnace operating at a temperature within the range between about 800° F. and about 1600° F. for a time period of between 15 seconds and 2 minutes roll compacting the dried strip by passing the same through a rolling mill while exerting a pressure ranging between about 500 and 3000 pounds per inch of strip width, normalizing the rolled compacted silicon steel strip in a furnace operating at a temperature within the range between about 1200° F. and about 1475° F. for a time period ranging between about 1 minute and about 4 minutes, and thereafter box annealing the normalized steel strip at a temperature within the range between about 2150° F. and about 2300° F. for a time period of from about 24 to about 60 hours.

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