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METHOD OF COATING SILICON STEEL IN CONJUNCTION WITH BOX ANNEALING THEREOF PREPARATORY TO DIE PUNCHING

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This invention relates to the separating medium coatings employed to prevent adhesion of adjacent wraps of steel strip during box annealing of the material in coil form, and is particularly concerned with removable coatings which do not impair die life in punching operations.

Typical of products requiring the application of a separating medium coating during production, and which will be subjected to die-forming operations, are grain-oriented silicon steel electrical sheets, which are fabricated into laminations of suitable shape to form the magnetic cores of electrical equipment, such as transformers and generators. In the production of such sheets, the steel is conventionally subjected to hot and cold rolling operations, which are followed by box annealing in coil form at a temperature of about 1800 to 2200° F., in hydrogen. A magnesia coating is usually applied to prevent adhesion of the adjacent wraps of the coiled material. This coating has been found to be the cause of premature wear of dies employed in fabrication of the material for its intended purpose. The magnesia coating, during the high temperature box anneal, forms a glassy-like iron-magnesium-silicate coating which impairs the punching quality of the sheet material, and is difficult to remove.

It is, therefore, an object of our invention to provide a coating that achieves the separating-medium protection required during box annealing, but may be completely removed thereafter.

Also, it is an object of our invention to prevent the formation of the aforementioned glass-like magnesium-silicate coating.

It is a further object of our invention to provide a pickling procedure for the substantially complete removal of said coating, after it has served its purpose, and prior to die-forming operations.

Coating mixtures containing magnesium oxide (MgO) and sodium chloride (NaCl), in which the latter is present in the proportion of about one (1) part in six (6) to one (1) part in twenty-five (25) of the weight of MgO, have been found highly satisfactory when applied in coating weights, of residual solids, of about 0.010 to 0.030 ounce per square foot of strip material.

Such coatings may be obtained from solutions in which the MgO ranges from about 5 to 15 ounces per gallon of water, preferably from about 7 to 11 ounces. At MgO contents substantially outside of these ranges it is more difficult, by usual procedures, to obtain resulting coatings of desired weights. At MgO contents substantially above about 10 ounces per gallon, the solutions tend to become difficult to handle, by virtue of their high viscosity.

It has been found that grain-oriented silicon steel, conventionally processed by rolling and annealing but employing the foregoing separating medium, may be cleaned of the medium by pickling in aqueous nitric-sulfuric acid mixtures. Satisfactory aqueous solution strengths range from about 10 to 20 percent by volume of concentrated nitric acid (sp. gr. 1.42 at 68° F.) with concentrated sulfuric acid (sp. gr. 1.84 at 68° F.) being at about 50% of the nitric acid, by volume.

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This solution is employed at about 85 to 110° F., preferably about 85 to 95° F., under which conditions coatings of this invention are completely stripped upon immersion for 30 to 90 seconds. Complete removal was obtained electrochemically in the same solution by pickling for 10 seconds anodic and 5 seconds cathodic at a current density within the range of 50 to 160 amperes per square foot, preferably about 100 amperes per square foot.

Some illustrative test results follow, wherein grain-oriented electrical sheets containing about 3.0 percent silicon received the following coatings:

Solution: ounces per gallon of distilled water		Coating weight applied: ounces per square foot of residual solids
MgO	NaCl	
10.2	1.1	0.010
10.2	0.5	0.015
10.2	1.6	0.020

The steel was then box annealed at a temperature of 2050° F. in a hydrogen atmosphere. The resulting coatings were examined by means of X-ray analysis and were found not to contain glass-like magnesium-silicate compound.

Samples of the aforementioned silicon steel, coated and annealed as outlined above, were submitted to the following pickling procedures:

(1) Samples coated with each mixture were immersed in a solution of 20 percent (vol.) HNO_3 and 10 percent (vol.) H_2SO_4 for 30 seconds at a temperature of 90° F.

(2) Identical samples were immersed in the same solution at the same temperature as (1) but were electrochemically pickled for 10 seconds anodic, 5 seconds cathodic at a current density of 100 amps. per square foot. In all of the above pickling tests the coatings were completely removed.

In punching tests under conditions where conventionally processed electrical sheets caused objectionable die burring in 1,000 to 3,000 punchings, sheets processed in accordance with the invention permitted approximately 80,000 punchings before an objectionable burr on the die resulted.

While specific examples and test results have been presented, these are to be understood as illustrative only, for purposes of ready comprehension and practice of the invention and not as limitations thereof or excluding such variations and modifications as would occur to one familiar with the art to which the invention appertains.

We claim:

1. In conjunction with box annealing of silicon steel strip preparatory to die punching, the steps of
   (a) coating said steel strip with a composition comprising essentially MgO and NaCl in which the NaCl is in the proportion of about one (1) part in six (6) to one (1) part in twenty-five (25) of the weight of MgO;
   (b) box annealing said coated strip; and
   (c) subsequently removing the coating resulting from said treatment by pickling in a composition comprising nitric and sulfuric acids.

2. The process of claim 1 in which the strip or the like is made successively anodic and cathodic during pickling.

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