Nov. 20, 1945. J. D. GAT 2,389,497

PRODUCTION OF ELECTRICAL SILICON STEEL

Filed April 14, 1943

FIG. 1.

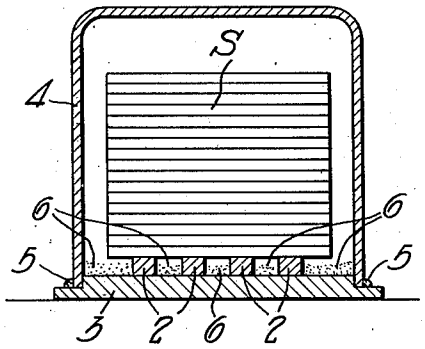


FIG. 2.

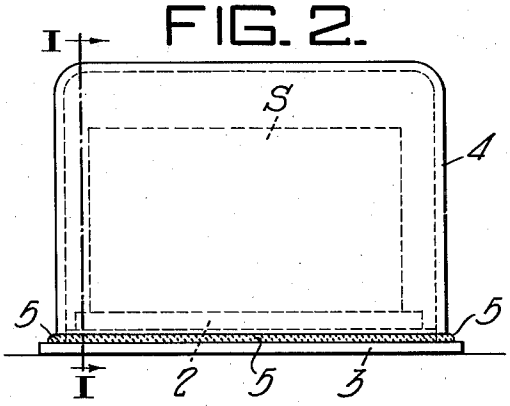


FIG. 3.

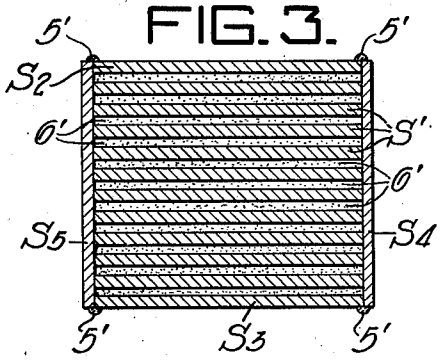


FIG. 4.

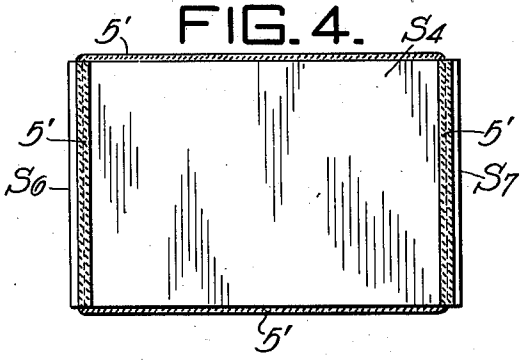


FIG. 7.

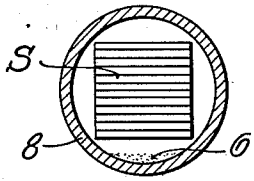
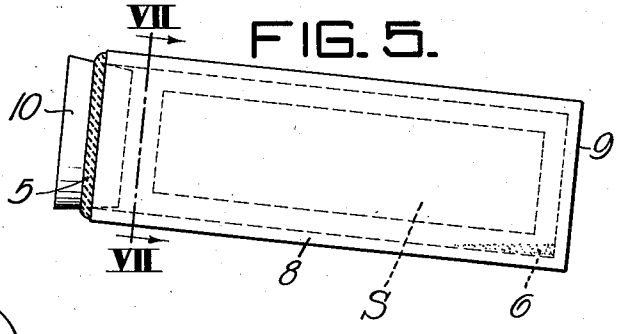


FIG. 5.



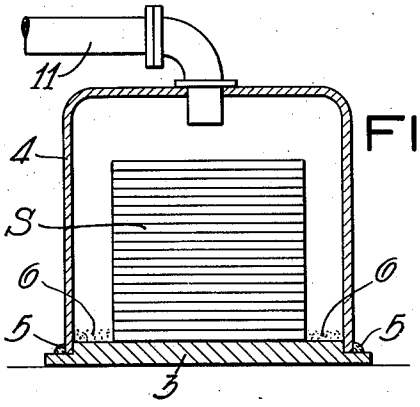


FIG. 6.

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UNITED STATES PATENT OFFICE

2,389,497

PRODUCTION OF ELECTRICAL SILICON STEEL

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Application April 14, 1943, Serial No. 483,021

1 Claim. (Cl. 148—21.5)

This invention is concerned with the production of silicon steel products having optimum electrical and mechanical properties. It is especially concerned with the heat treatment of electrical silicon steel sheets and strip in such a manner as to alter the composition thereof to the benefit of its properties.

In accordance with established steel making and processing practices, it is inevitable that silicon steels have, in addition to the expected contents of iron, silicon and carbon, quantities of additional elements in varying amounts, which are largely non-metallic impurities remaining in the steel after its refinement in the steel making process, or acquired during subsequent heat treatment or hot rolling. Carbon itself, though expected in small proportion in steels of this class, is frequently too high for the attainment of the desired electrical values and mechanical properties. Other non-metallic elements frequently found in such steels, either in combined or uncombined form, are oxygen, nitrogen, hydrogen, sulphur and phosphorus.

For the attainment of optimum electrical properties, it is generally regarded that silicon steels should, as far as practicable, consist of pure iron and silicon, and should have homogeneity, perfection of crystallinity, and freedom from all strain. Such an ideal alloy could, by a minimum amount of treatment in an appropriate manner, be invested with optimum electrical and mechanical properties by the establishment in the metal of the essential grain condition, as to size and orientation, and by maintaining the steel completely free of such impurities as are antagonistic to the attainment thereof.

Since the presence of non-metallic elements in steel produced by commercial methods is unavoidable, processing must be adopted with a view toward, either: (1) disposing such impurities in the metal in such chemical and physical condition as to minimize their harmful effects, or (2) eliminating them as completely as possible, thus removing them from consideration. Considerable work has been undertaken in the processing of the electrical silicon steels so as to achieve both of these ends, although, by virtue of the fact that steels placed in such condition as readily to give up the impurities are, by the same token, susceptible to recontamination by the same, or equally objectionable, elements, the tendency has been toward the former course rather than the latter.

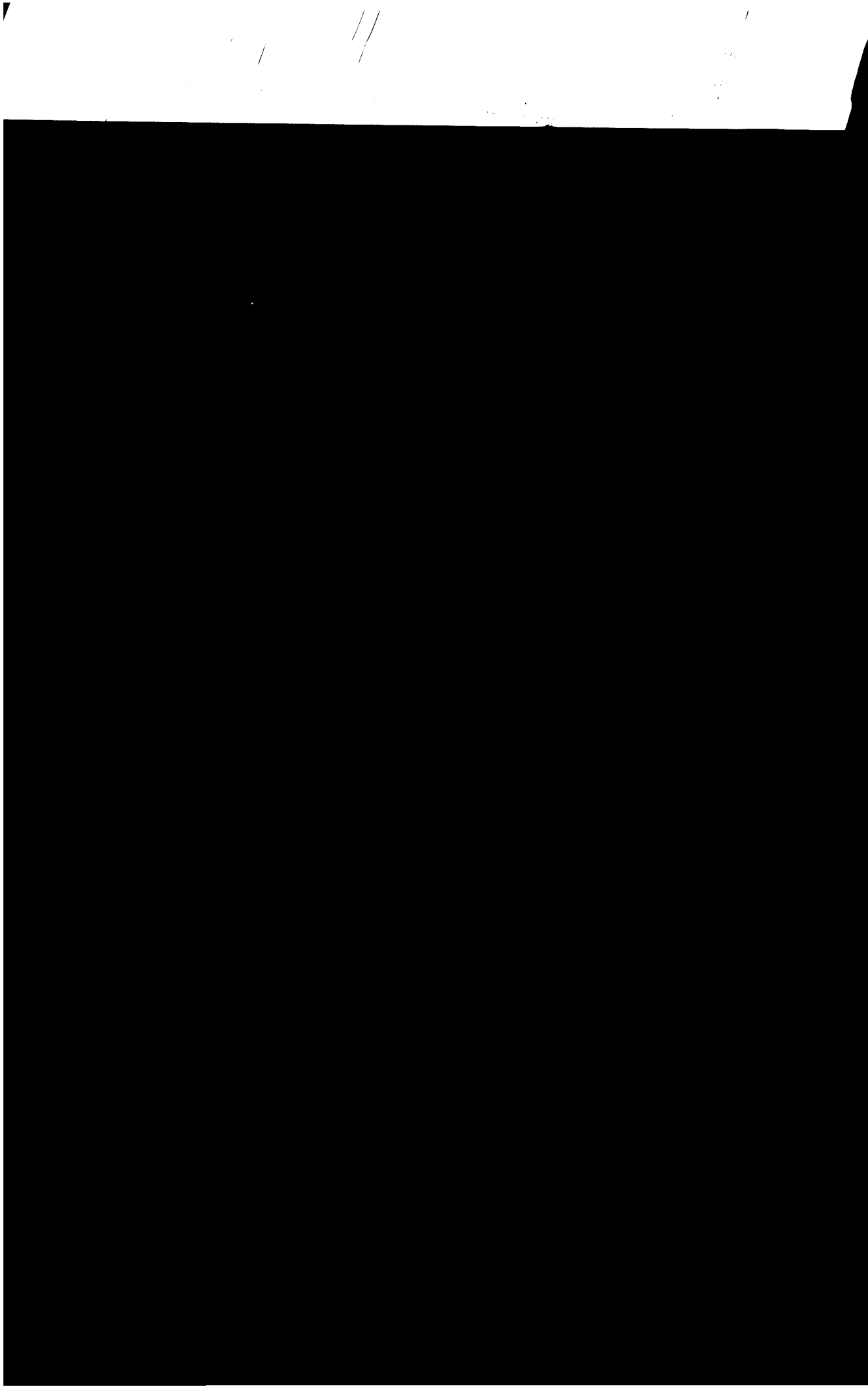
The present invention is directed toward the treatment of electrical silicon steels in such a manner as to cause the latter to give up those non-metallic elements, which are present in such amounts as to impair the electrical and mechanical properties, to the extent that such elements are either eliminated, or greatly lessened in their effective amounts, resulting in curing the steel of substantially all deleterious effects attributable to their presence.

Carbon is generally regarded as harmful to the electrical and magnetic properties. Present in very low concentration, it distorts space lattice of silicon ferrite with the subsequent deleterious effect on magnetic properties of the metal. In greater amounts, the carbon tends to form austenite at higher temperatures. Upon cooling to room temperature, the original austenitic areas result in ferrite of a more or less distorted nature, the presence of which is always undesirable in magnetically efficient alloys. This phenomenon cannot be prevented by the application of a controlled cooling rate tending toward the establishment of equilibrium conditions in the metal. Neither can the effect of carbon be remedied by thermal cycles tending to precipitate cementite in shape of graphite. Though under such conditions the space lattice distortion can be eliminated practically completely, the presence of discrete graphite particles reduces the effective cross section of the metal resulting in corresponding loss of electrical characteristics.

The iron-nitrogen system corresponds closely to the iron-carbon system, just discussed, and nitrogen, like carbon, is regarded as being a very serious contaminating influence in electrical steels even in minute quantities.

Oxygen is one available reagent by which carbon may be removed from steels of this class, or rendered in a fairly stable chemical form in which its bad effects are minimized. However, oxygen in uncontrolled amounts not only excessively oxidizes steels which are exposed at high temperature to its influence, but combines with the silicon content, as well as with any other active metallics present, such as aluminum, to form the oxides thereof, in which condition they are not useful or active as in their uncombined form, and in which condition they further provide "dirt" throughout the steel, which, in microscopic and submicroscopic condition, can cause a very serious impairment of the electrical properties.

The metalloids—sulphur and phosphorus—are well-known for their deleterious influences on the mechanical properties of steel in general, and are no more desirable from the electrical standpoint. The sulphur compounds of iron increase dielectric and lower magnetic character-



accordance with the present invention, however, the getters, by being effective in lowering the partial pressure of the deleterious gases, cause a uniform and well-distributed movement of the latter from whatever may be their source of origin interiorly or exteriorly of the charge. This, of course, has reference to a partial pressure of elements and their compounds, rather than actual atmospheric pressure, which, in some cases, may even be higher externally of the charge than internally. It is this partial pressure phenomenon that renders the sealing of the more open types of furnaces extremely difficult, in so far as the exclusion of those gaseous elements active toward the metal under treatment is concerned. By hermetically sealing the charge, all influences, except those within the container, are eliminated, rendering the getter free to effect lower partial pressure of the substances with which it combines, thus to effect uniform transmigration of such substances from an entire charge, and, thus, to insure absolutely uniform results throughout the entire mass under treatment, irrespective of its placement in the container.

From the foregoing, it will be obvious that the material selected for the container, or annealing receptacle, should be of such composition as will not bring an undue amount of contaminating influences to the enclosed atmosphere, since this would impose an undue tax upon the capacity of the getter, and cause the requisite quantity of the latter, as well as the time necessary for the effective treatment of the charge, to become excessive. Thus, the container should not be high in those nonmetallic constituents to which the getter is active, which it is desired to remove from the enclosed charge. In fact, the container may be composed of some material which will in itself, act as a getter, such as stainless steel, or other alloy steels high in active ingredients. In the latter case, the container may serve as the only getter substance present during the heat treatment, although it is more usual to include an additional active substance within the container, relative to which the container metal, per se, acts in a complementary relation.

In the attached drawing, several unrestrictive forms of apparatus for giving effect to the present invention are illustrated, as follows:

Figure 1 illustrates one of the more conventional forms of annealing receptacles, for accommodating a stack of sheets in accordance with the present invention, shown in cross-sectional elevation taken along line I—I, Figure 2.

Figure 2 represents a side elevational view of the container shown in Figure 1.

Figure 3 is a view corresponding to Figure 1 of another manner of hermetically sealing a charge of sheets in accordance with this invention.

Figure 4 is a side elevational view of the arrangement shown in Figure 3.

Figure 5 is a side elevational view of another form of annealing receptacle made in accordance with the present invention.

Figure 6 is a view corresponding to Figure 1 of a similar receptacle provided with evacuating means.

Figure 7 represents a cross-sectional elevational view taken along line VII—VII in Figure 5.

Referring more specifically to the drawing, in Figures 1 and 2, a charge comprised of a stack of sheets S is supported by suitable spacer bearings 2 upon the base 3 of an annealing container. A cover 4 is arranged completely to cover the charge, and closely to cooperate with the base, so as to form a hermetic seal therewith, as by a continuous welded seam 5. In these figures, the getter substance 6 has been represented as a powder distributed across the floor of the container beneath the charge. It is obvious that any suitable disposition of the getter may be provided, as by wall pockets or holders (not shown) arranged therefor on the inner walls of the cover 4.

In Figures 3 and 4, the charge is represented as a stack of sheets S′ sandwiched with layers of the getter substance 6′, so as to interpose the getter between each of the sheets, as shown in Figure 3. In this arrangement, the getter serves an additional function of maintaining the sheets in spaced relation to prevent sticking thereof at high temperatures, and, thus, should be a nonfusible substance to prevent sintering and sticking of the sheets together. Calcium hydride, preferably mixed with an inert filler material, e. g., lime, magnesium oxide, or talc, is an example of one suitable material for this purpose. In this embodiment, the top sheet S_2, and the bottom sheet S_3, cooperate with suitably disposed vertical sheets S_4, S_5, S_6 and S_7, constituting the sides and ends, respectively, of the container. These six last named sheets are all suitably joined together as by welds 5′ to constitute an hermetic enclosure.

In the embodiment shown in Figures 5 and 7, a tube 8, having a bottom closure 9 integrated therewith, is provided with a tapered plug 10 at its open end, which, after the charge S has been introduced therein, is driven into place and welded as at 5. A getter substance 6 is disposed in any suitable manner within the tube. Inasmuch as this frequently takes the form of some of the metallic substances, such as, magnesium, aluminum, etc., above the melting point of which the charge is heated, it is desirable to tilt the tube slightly, as indicated in Figure 5, in order that the getter substance in liquid form will not run between the sheets and tend to braze them together.

In the modification shown in Figure 6, there is an annealing receptacle, similar to that shown in Figure 1, comprising a base 3, and a cover 4, united to the latter, as by a welded seam 5. An evacuating tube 11 is provided at any suitable location, through which the gaseous contents of the receptacle may be withdrawn. Such an arrangement may be regarded as typical for any of the forms of annealing receptacles here shown, and is not restricted to the box type shown in Figures 1, 2 and 6.

Apart from the evacuating means, of which the device shown in Figure 6 is representative, any of the hermetic containers here illustrated my be evacuated, or partially evacuated, before being hermetically sealed at the outset. Means for such purpose are old and well-known to the art, and need not be illustrated. Also, any suitable hermetic receptacle is contemplated, whether for the accommodation of stacks of sheets, or coils of strip, and the invention is not limited to the specific forms of apparatus shown in the drawing.

Silicon steels treated in accordance herewith have manifested substantial reductions in the quantities of non-metallic elements present; particularly, carbon, nitrogen, oxygen and sulphur, and have proved to possess electrical and mechanical properties for any given grade (determined by the silicon content) corresponding to those for higher grades having greater silicon

content but with the superior mechanical properties of the lower silicon grades unaffected. It is, thus, possible to obtain improved electrical and mechanical properties with less silicon than has heretofore been possible, which considerably simplifies the processing operations; particularly, those of reduction to gauge and heat treatment. In this latter connection, it is known that for the attainment of optimum electrical properties, the higher the silicon content, the higher must be the heat treating temperature, unless special alloying additions are included, such as aluminum. The attainment of the properties of the higher grades at lower silicon contents results, therefore, in considerably simplified processing, and the full development of the electrical and mechanical properties in one heat treatment conducted in accordance with the principles here set forth.

Specifically to illustrate the results recited in the foregoing paragraph, the following demonstrations have been performed:

Charges of silicon steel sheets that had been reduced to gauge, pickled, cold rolled to flatten, and coated with a sticker prevention coating, were sealed in hermetic containers, so that the weight of sheets in each approximated 23½ tons. Sealed within each of the containers were fifteen pounds of a getter composition; in one case comprising ten pounds of aluminum and five pounds of magnesium, and in another case comprising ten pounds of aluminum and approximately two and one-half pounds each of magnesium and calcium. The containers, thus charged, were sealed by careful welding, and were disposed in the furnace, and heated until the charge attained a temperature of approximately 1950° F. The results as to core loss at an induction of 10,000 gausses in relation to the silicon content were as follows:

Box 115 aluminum-magnesium getter

	Silicon content	Core loss 10,000 B
1	3.60	.559
2	3.60	.569
3	3.79	.557
4	3.79	.569
5	3.79	.570
6	3.79	.577
7	4.39	.532

Box 116 aluminum-magnesium-calcium getter

	Silicon content	Core loss 10,000 B
8	3.60	.541
9	3.79	.518
10	3.79	.536
11	3.79	.539
12	4.56	.495

The above watt loss values for the silicon contents given indicate that these steels exhibited magnetic properties, in consequence of the heat treatment, corresponding to those higher grades of steels containing from 4.15 to 4:45 per cent silicon. It is interesting to note that the sheets listed under No. 8, above, showed a core loss of a low value usually associated with silicon steels nearly two grades higher than the 3.60 silicon steel here exhibited. It is also interesting to note that the material listed under No. 7, above, was from a previously conventionally treated run of sheets, which, though having satisfactory core loss values, showed such marked impairment of ductility, as to have been rejected as being nonsalably brittle. Upon retreatment, in accordance with the present invention, as given above, the ductility was restored to a suitable value, thus demonstrating the effectiveness of the present invention in perfecting the mechanical, as well as the electrical, properties of steels of this class. It also may be of interest to note that some 900 grams of carbonaceous residue, analyzing 25% carbon, were collected within the container, which had deposited therein remote from the charge, and in the proximity of the getter, particularly the magnesium portion thereof.

It is within the scope of the present invention to utilize a getter substance, which, after reacting with the deleterious ingredients, may give off a gas to replace the internal atmosphere of the container as a reaction product. In this particular, those compounds which yield hydrogen, such as calcium hydride, titanium hydride, etc., provide a hydrogen atmosphere which acts as a flushing gas. Or, if desired, a suitable flushing gas may be introduced through an evacuating outlet after evacuation of the included gases. The presence of hydrogen renders the enclosed atmosphere both reducing and decarburizing, and, thus, results in the appearance of water and hydrocarbon gases within the container. The moisture content, the hydrocarbon gases, such as methane (CH_4), and the carbon oxide gases (CO and CO_2), because of their ready dissociation and activity at the heat treating temperatures, must be subject to reaction and stabilization by the getter substances employed, as has already been described herein, above.

Thus, the present invention contemplates the removal from the silicon steel charge: (1) of carbon, principally as a gas in the form of the carbon oxides, or as a hydrocarbon; (2) of oxygen as free oxygen, or as combined with carbon (CO and CO_2), and hydrogen (H_2O); (3) of nitrogen as free nitrogen, or as ammonia, or cyanogen gas. In addition, a marked reduction of manganese has been observed from these hermetic annealing practices in the presence of getters, as well as that of sulphur, and, to a less extent, phosphorus, which, by virtue of the fact that most of these steels are open hearth steels, is usually of a very low order in the first instance.

It will be understood, therefore, that many modifications of the invention may be made without departing from the scope thereof, and it is not intended that I be limited to the specific embodiment shown, other than as is rendered necessary by the recitations of the appended claim.

I claim:

In the processing of electrical silicon steel sheets and strip, the improvement which includes hermetically sealing such steel within an enclosure in the presence of a substance active at elevated temperatures toward the nonmetallic elements within said enclosure, and heating the steel and said substance sufficiently to effect diffusion of the nonmetallic elements through the steel, said method further including evacuating the atmosphere from the enclosure incident to the hermetic sealing thereof.

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