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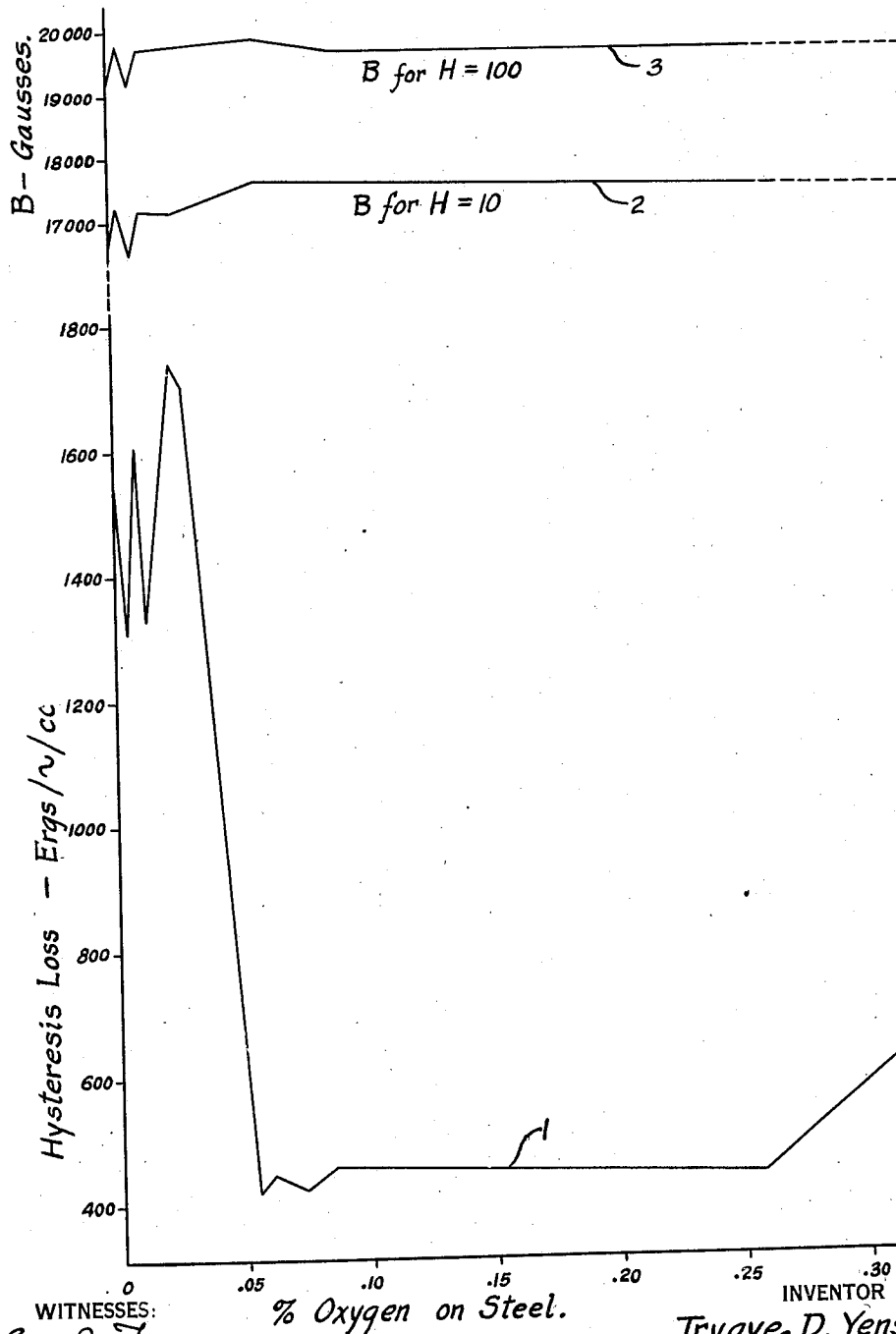
T. D. YENSEN

2,050,408

PROCESS OF TREATING MAGNETIC MATERIAL

Filed Oct. 23, 1935

5 Sheets-Sheet J



WITNESSES:

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% Oxygen on Steel.

Fig. 1.

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2,050,408

PROCESS OF TREATING MAGNETIC MATERIAL

Filed Oct. 23, 1935

5 Sheets-Sheet 2

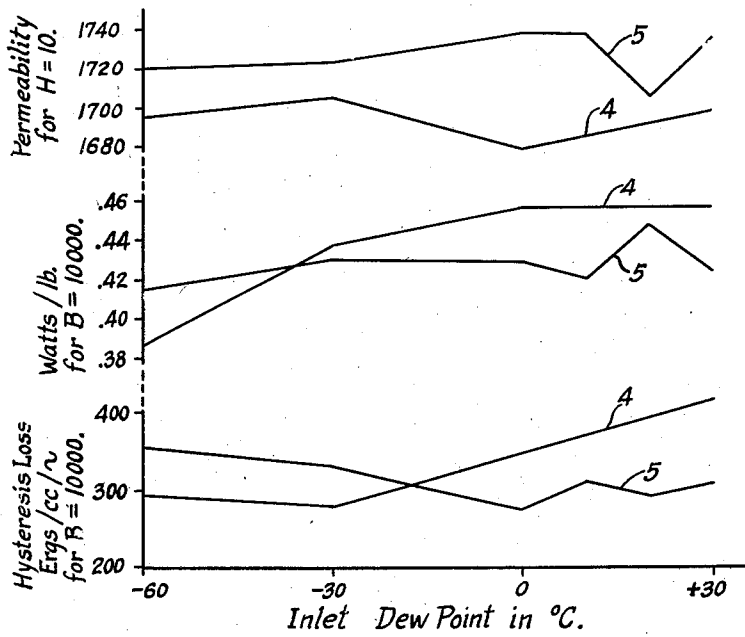


Fig. 2.

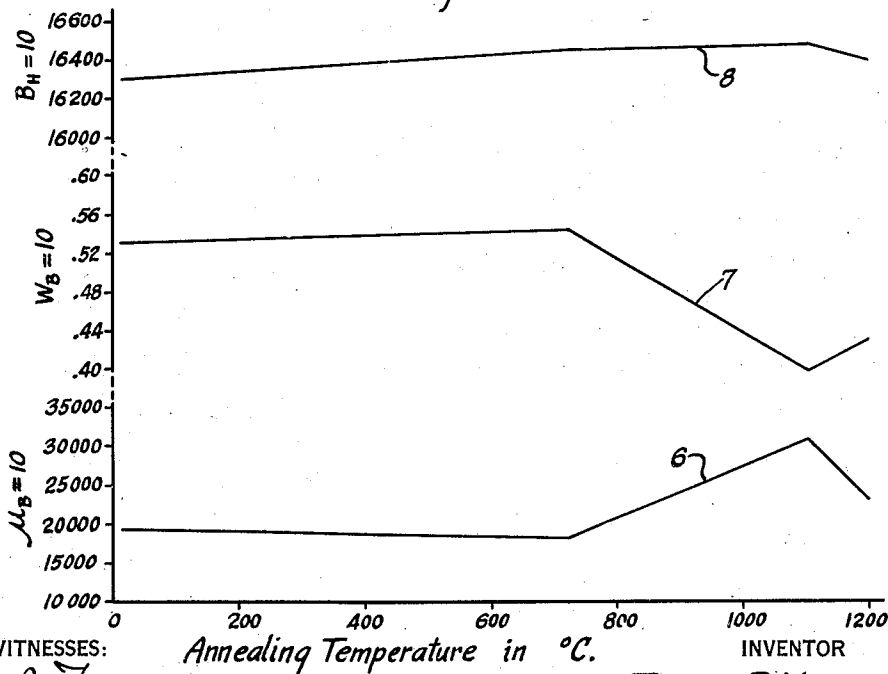


Fig. 3.

WITNESSES:

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Annealing Temperature in °C.

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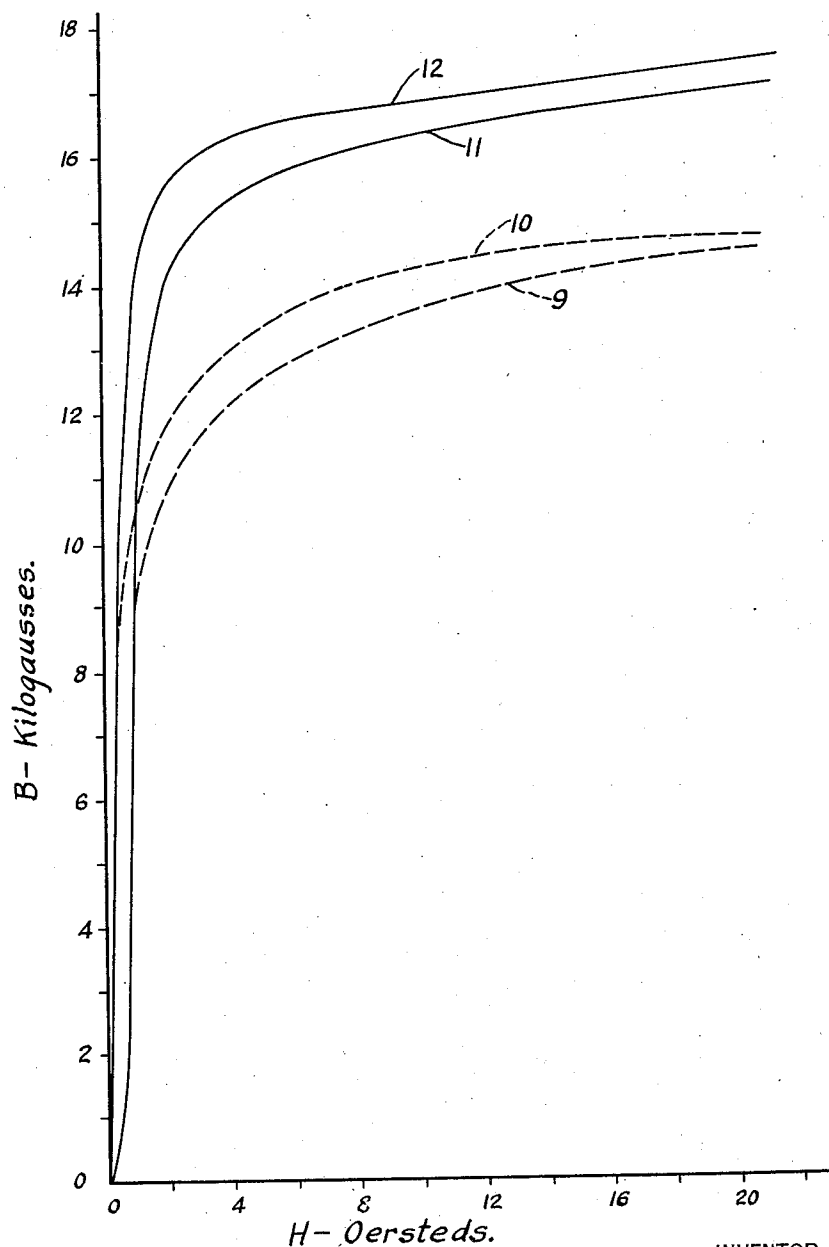
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Filed Oct. 23, 1935

5 Sheets-Sheet 3



WITNESSES:

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Fig. 4.

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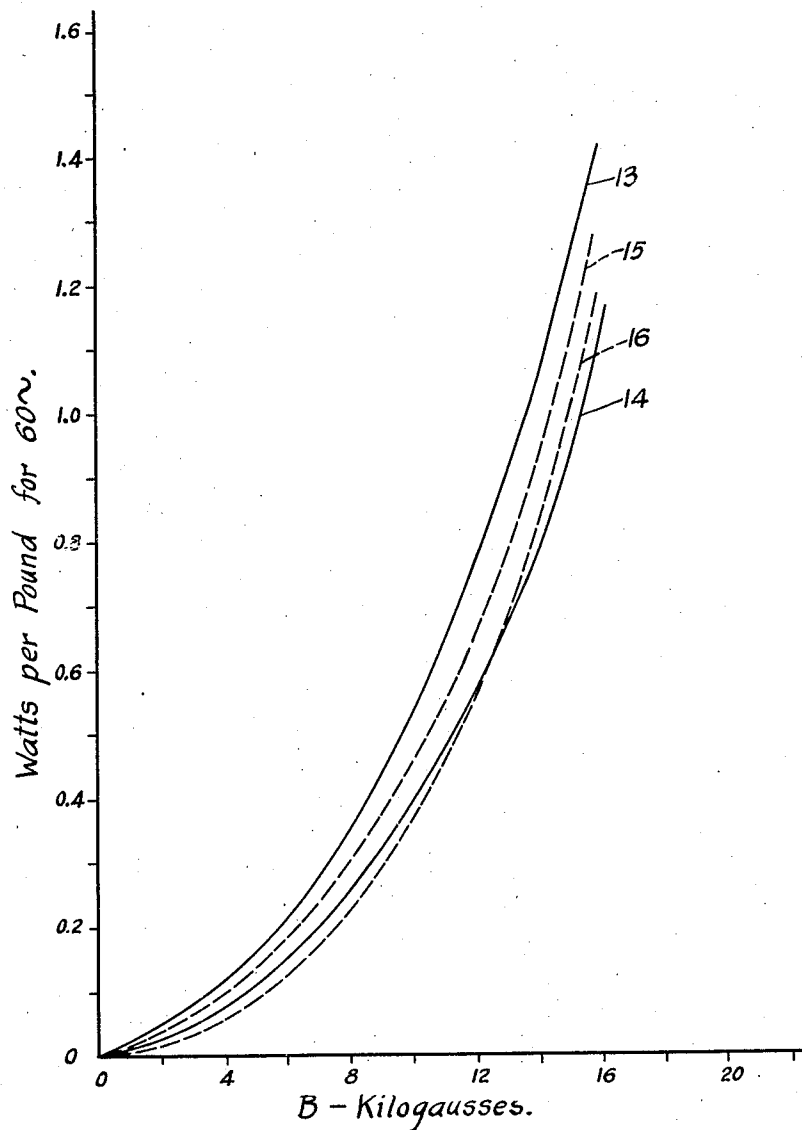


Fig. 5.

WITNESSES:

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Aug. 11, 1936.

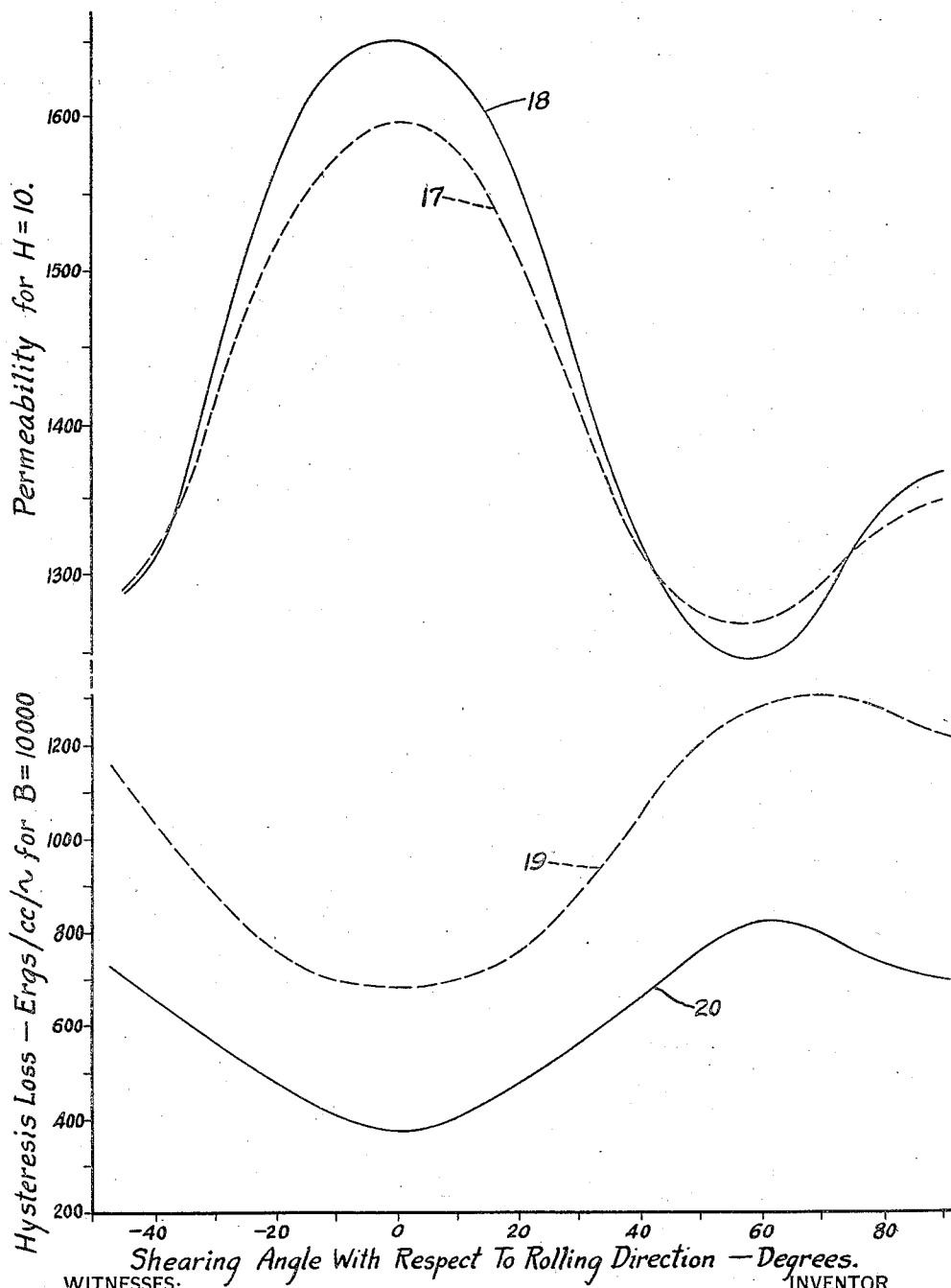
T. D. YENSEN

2,050,408

PROCESS OF TREATING MAGNETIC MATERIAL

Filed Oct. 23, 1935

5 Sheets-Sheet 5



WITNESSES:

Leon J. Taza.
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Fig. 6.

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UNITED STATES PATENT OFFICE

2,050,408

PROCESS OF TREATING MAGNETIC MATERIAL

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Application October 23, 1935, Serial No. 46,349

7 Claims. (Cl. 148—14)

This invention relates to the heat treatment of magnetic material and particularly to the process of heat treating silicon iron.

5 An object of this invention is to provide a process for heat treating silicon iron for developing its desirable magnetic properties.

10 Another object of this invention is to provide for heat treating silicon iron to reduce impurities therein and develop its desirable magnetic properties.

15 A further object of this invention is to provide for removing the impurities from silicon iron and increasing the preferred orientation of the material to develop its desirable magnetic properties.

20 This invention, together with other and additional objects, may be better understood from the following description taken in conjunction with the accompanying drawings, in which

Figure 1 is a graph, the curves of which show how oxygen affects the magnetic properties of silicon iron when heat treated in accordance with this invention.

25 Fig. 2 is a graph showing how the moisture content and the rate of flow of hydrogen affects the desirable magnetic properties of silicon iron when heat treated in accordance with this invention.

30 Fig. 3 is a graph, the curves of which show the effect of different annealing temperatures on the desirable magnetic properties of silicon iron which has been treated in accordance with this invention.

35 Figs. 4 and 5 are graphs showing the improvements in the desirable magnetic properties of silicon iron which has been heat treated in accordance with the teachings of this invention; and

40 Fig. 6 is a graph, the curves of which show the improvement in the preferred crystal lattice orientation imparted to silicon iron as measured in terms of the permeability and the improvement in the hysteresis losses in silicon iron when magnetized in different directions relative to the rolling direction after the iron has been heat treated in accordance with the teachings of this invention.

45 In the electrical industry, magnetic material such as silicon iron is employed in the manufacture of different electrical apparatus. Many types of silicon iron suitable for such use are available on the market. These commercial irons have good magnetic properties, combining low hysteresis losses with high permeability values. It has been found possible to purify silicon iron now com-

mercially sold to such a degree that its desirable magnetic properties are further developed.

In preparing the silicon iron for use in the different electrical apparatus, it has been found possible to lower the impurity content of the iron below that normally present in commercial iron and to increase the desirable magnetic properties of the iron by subjecting it to the heat treatment embodied in this invention. This heat treatment is of special value when applied as a further heat treatment to commercial silicon iron in which preferred orientation has been developed, since the heat treatment of this invention further increases the preferred orientation and consequently increases the desirable magnetic properties of the iron.

In practicing this invention, silicon iron containing up to approximately $4\frac{1}{4}\%$ silicon which has been previously heat treated to develop its desirable magnetic properties is subjected to a further heat treatment to lower the carbon content, increase the preferred orientation and increase the desirable magnetic properties of the iron.

In preparing silicon iron which has been previously heat treated for the process embodied in this invention, sheets of the silicon iron are coated with a refractory to prevent the sticking of the sheets when stacked and subjected to the heat treatment. The refractory further separates the sheets to permit the reducing gas employed in the process to completely envelop each of the sheets. In order to be satisfactory, the refractories must also be sufficiently permeable to permit ready diffusion of hydrogen and carbon monoxide gases. Refractories, such as aluminum oxide, magnesia, silica, flint and talc, have been found to meet all the conditions imposed thereon.

40 The refractory may be applied to the iron in the form of a paint prepared by grinding the refractory in a ball mill and mixing it with water, alcohol or any other suitable liquid to a paint-like consistency, after which it may be applied to the sheets by brushing, spraying or dipping.

45 In order to reduce the carbon content of the iron and increase its magnetic properties, it has been found necessary to provide sufficient oxygen on the surface of the iron to react with the carbon which normally diffuses to the surface of the iron during the heating. The reaction products, carbon oxides such as carbon monoxide and carbon dioxide, thus formed are removed by the flow of hydrogen employed in the heat treatment. The refractory employed on the strip

does not of itself provide sufficient oxygen to react with the carbon and it is, therefore, necessary to provide for oxidizing the surface of the iron prior to the anneal.

5 The oxygen employed for reacting with the carbon of the iron may be applied directly to the surface of the iron in the form of an iron oxide (Fe_2O_3) which has been prepared by grinding the oxide into a fine form. The iron oxide is then brushed or sprinkled over the surface of the iron, after which the refractory chosen may be applied to the iron oxide coated strips. If desirable, paint comprising a mixture of the iron oxide and refractory may be applied directly to the surface of the iron.

15 It has been proven by experiments that sufficient oxygen on the surface of the iron is necessary for reacting with the carbon content of the iron during the heat treatment to reduce the carbon content to less than .005%. A satisfactory coating for accomplishing this result has been found to be one containing from .05% to .3% by weight oxygen.

25 The results of a series of tests showing the effect of different oxygen contents on the surface of the iron on the desirable magnetic properties of silicon iron treated by this process are plotted in Fig. 1 of the drawings.

30 Curve 1 of Fig. 1 shows the effect of the different oxygen contents on the resulting hysteresis losses and clearly indicates that a surface coating containing from .05% to .30% by weight of oxygen is to be preferred. The lowest hysteresis losses are produced where the oxygen content is within this range. Where less than .05% of oxygen is employed on the surface of the iron, the hysteresis losses are undesirably high. The effect of the different oxygen contents on the flux densities for magnetizing forces of 10 and 100 oersteds is shown by curves 2 and 3, respectively, of Fig. 1.

40 Where the oxygen is not applied to the surface of the iron in the form of iron oxide, it is desirable to oxidize the iron by some method suitable for supplying sufficient oxygen to react with the carbon content of the iron. This may be accomplished by applying the refractory in a wet form to the iron and allowing it to dry in air for a sufficient length of time to cause the surface of the iron to rust.

50 Other methods which have been found to be satisfactory are to apply the refractory to the silicon iron and heat it at a temperature of 500°C . in air for a period of about two hours or to heat the refractory coated iron in a gas furnace at a temperature of 500°C . In all cases, the amount of oxygen deposited on the surface of the iron may be controlled so as to deposit from .05% to .3% by weight oxygen on the surface of the iron.

60 After the silicon iron has been sufficiently oxidized, it is subjected to a heating temperature of between 900°C . and 1300°C . for a period of from 10 to 20 hours. The heating is conducted in a reducing atmosphere comprising hydrogen, the flow and moisture content of which is closely controlled.

65 In order to prevent further oxidation of the silicon iron during the heat treatment, it has been found desirable to control the moisture content of the hydrogen employed as the reducing atmosphere. The moisture content of the hydrogen has been found to be of little consequence where a low rate of flow of hydrogen is employed, but it has been found that with a high rate of flow of hydrogen, the moisture content should be low in order not to cause excessive oxidation of the

iron and thereby not only remove a portion of the silicon content but also depreciate the desirable magnetic properties.

Experiments have proved that hydrogen having a dew point of from -60°C . to $+30^\circ\text{C}$. may be satisfactorily employed where the rate of flow of the hydrogen is about 1.75 liters per minute per kilogram. Where a higher rate of flow of hydrogen, such as 16 liters per minute per kilogram is employed, the experiments have proved that the dew point of the hydrogen should be between -60°C . and zero degrees C.

By reference to Fig. 2 of the drawings, the combined effect of the moisture content and the rate of flow of the hydrogen in the furnace during the heat treatment of this invention on the desirable magnetic properties of the silicon iron is evident. As viewed in the drawings, curves 4 illustrate the effect of the dew point with a high rate of flow of hydrogen, 16 liters per minute per kilogram, on the desirable magnetic properties of iron treated in accordance with this invention, while curves 5 illustrate the effect of the same dew points for a low rate of flow of hydrogen, 1.75 liters per minute per kilogram, on the desirable magnetic properties of silicon iron that has been given a similar heat treatment. From these curves, it is evident that with a high rate of flow of hydrogen it is desirable to maintain a low moisture content. In practice, a hydrogen flow of between 0.1 and 3 liters per minute per kilogram with a dew point of from -60°C . to -30°C . has proved to be satisfactory.

Although the magnetic properties of the silicon iron may be greatly improved by heating it in hydrogen at a temperature of from 900°C . to 1300°C ., it has been found that best results may be obtained by heating it at temperatures of between 900°C . and 1100°C . When heating at temperatures of 1100°C . or lower, the oxygen on the surface of the iron reacts with the carbon which diffuses to the surface of the iron to remove it as an impurity in the flow of the hydrogen gas. At temperatures higher than 1100°C ., the oxide coating on the iron has been found to be reduced before it has had sufficient time to combine with the carbon which normally diffuses to the surface of the iron. Satisfactory results may, however, be obtained at temperatures of 1200°C . when the oxygen content on the surface of the iron and the moisture content and rate of flow of the hydrogen are closely controlled.

70 In Fig. 3 of the drawings, curves 6, 7 and 8 represent the permeabilities, watt loss, and flux density, respectively, of silicon iron prepared as hereinbefore described and heat treated at different temperatures in a controlled hydrogen atmosphere. The curves clearly illustrate that best results are obtained by heating the magnetic material at temperatures of about 1100°C .

75 Figs. 4, 5 and 6 of the drawings illustrate the remarkable improvement in the desirable magnetic properties of silicon iron which has been previously heat treated to develop its magnetic properties, and then subjected to the heat treatment embodied in this invention.

In Fig. 4, the flux density values in kilogausses are plotted against the magnetizing force in oersteds for two types of commercially heat treated silicon iron which have been further heat treated in accordance with the teachings of this invention to lower the impurity content and further increase the permeability. In Fig. 4, curve 9 represents a $4\frac{1}{4}\%$ silicon iron that has been previously subjected to a hot-rolling and a heat

treatment to develop its magnetic properties, while curve 10 represents the improvement in its desirable magnetic properties when subjected to the heat treatment of this invention. Curve 11 represents a 3¼% silicon iron that has been previously subjected to a cold-rolling and a heat treatment, while curve 12 shows the improvement in the desirable magnetic properties in the same iron when subjected to the further heat treatment hereinbefore described. The same heat treatment, namely, annealing the oxidized silicon iron in a controlled hydrogen atmosphere at a temperature of 1200° C. for ten hours was accorded each of the two types of commercially heat treated iron.

In Fig. 5, the watts loss at different flux densities for the cold-rolled and hot-rolled iron heat treated and identified in the discussion of the curves of Fig. 4 are plotted to show the improvement in the losses of the iron when subjected to this heat treatment. In Fig. 5, curves 13 and 14 represent the losses developed in the cold-rolled iron at different flux densities and curves 15 and 16 represent the losses developed in the hot-rolled iron at corresponding flux densities. The reduction of losses in the iron after it has been subjected to the heat treatment of the present invention is evident from an examination of these curves.

As stated hereinbefore, the heat treatment of this invention is of special value when applied to silicon iron in which preferred orientation has been developed since the heat treatment further increases the preferred orientation and consequently increases the desirable magnetic properties of the iron. An examination of the curves of Fig. 6 of the drawings will reveal that such improvement in the preferred orientation is desirable in further developing the magnetic properties of the iron. In Fig. 6, curve 17 represents the permeability values of a commercial 3¼% silicon iron which has been given a previous heat treatment to develop the preferred crystal lattice orientation plotted against the shearing angle of the iron with respect to the rolling direction. Curve 18 represents the permeability values of the same iron after it has been subjected to the further heat treatment of this invention. As indicated by the curves, remarkable improvement in the desirable magnetic properties of the iron is found in the direction of the preferred orientation.

In Fig. 6, curves 19 and 20 represent the hysteresis losses at a flux density of 10,000 for a 3¼% silicon iron, as measured before and after being subjected to the heat treatment of this invention, plotted against the shearing angle with respect to the rolling direction of the iron. In the figure, curve 19 represents the losses in the iron after it has been subjected to the usual commercial heat treatment, while curve 20 represents the losses in the same silicon iron after it has been subjected to the further heat treatment of this invention. As indicated by the curves, the hysteresis loss has been lowered 300 ergs per c. c. per cycle for B=10,000 at the 0° shearing angle. Corresponding improvements in the losses are found with respect to the different shearing angles.

As is evident from the examination of the curves in the different figures hereinbefore described, silicon iron, which has been heat treated as hereinbefore set forth, has its desirable magnetic properties developed to an unusual extent. In silicon iron heat treated as herein de-

scribed, core loss values of the order of .72 to .84 watts per kilogram at 60 cycles and 10,000 lines per square inch, have been consistently secured for sheets of iron ranging from .011 to .014 inches thick. Further, consistent permeability values at high inductions may be secured. As a specific example of the values which may be secured, a 3¼% silicon iron strip .0135 inches thick, after being heat treated in accordance with this invention, had a hysteresis loss for B=10,000 of 300 ergs per c. c. per cycle with a permeability of 1750 for H=10 and a permeability of 195 for H=100 and a watts loss for B=10,000 of .75 watts per kilogram at 60 cycles and 2.20 watts per kilogram at 60 cycles for B=16,000.

In addition to the good magnetic properties developed in the silicon iron, it is found that they are ductile and are easy to punch into the shapes desired by the industry.

Although this invention has been described with reference to a particular heat treatment, it is, of course, to be understood that other and various modifications thereof may be possible. This invention is therefore not to be restricted except insofar as is necessitated by the prior art and the scope of the appended claims.

I claim as my invention:

1. In the process of heat treating silicon iron, in combination, coating the silicon iron with a refractory, oxidizing the coated iron to deposit from .05% to .3% by weight oxygen over the surface of the iron, heating the silicon iron at a temperature of from 900° C. to 1300° C. in hydrogen to decarbonize the iron, the hydrogen having a low moisture content and a rate of flow of between 0.1 and 16 liters per minute per kilogram, and cooling the decarbonized iron in hydrogen to a temperature of below 500° C.

2. In the process of heat treating silicon iron, in combination, coating the silicon iron with an iron oxide to deposit from .05% to .3% by weight oxygen over the surface of the iron, applying a refractory over the iron oxide coated iron to prevent sticking of the iron when stacked and heated, heating the silicon iron at a temperature of from 900° C. to 1300° C. in hydrogen to decarbonize the iron, the hydrogen having a low moisture content and a rate of flow of between 0.1 and 16 liters per minute per kilogram, the carbon content of the iron being reduced to less than .005%, and cooling the decarbonized iron in hydrogen to a temperature of below 500° C.

3. In the process of heat treating silicon iron, in combination, coating the silicon iron with a refractory, oxidizing the coated iron to deposit from .05% to .3% by weight oxygen over the surface of the iron, heating the silicon iron at a temperature of from 900° C. to 1300° C. in hydrogen for from 10 to 30 hours to decarbonize the iron, the hydrogen having a low moisture content and a rate of flow of between 0.1 and 3 liters per minute per kilogram, the carbon content of the iron being reduced to less than .005%, and cooling the decarbonized iron in hydrogen to a temperature of below 500° C.

4. In the process of heat treating silicon iron, in combination, coating the silicon iron with a refractory, oxidizing the iron to deposit from .05% to .3% by weight oxygen over the surface of the iron, heating the silicon iron at a temperature of about 1100° C. in hydrogen for from 10 to 30 hours to decarbonize the iron, the hydrogen having a low moisture content and a rate of flow of hydrogen of less than 2 liters per minute per kilogram, the carbon content of the iron being 75

reduced to less than .005%, and cooling the decarbonized iron in hydrogen to a temperature of less than 500° C.

- 5 In the process of heat treating silicon iron, in combination, coating the silicon iron with a refractory, oxidizing the coated iron to deposit from .05% to .3% by weight oxygen over the surface of the iron, heating the silicon iron at a temperature of from 900° C. to 1300° C. in hydrogen for from 10 to 30 hours, the hydrogen having a dew point of from -60° C. to 0° C. when the rate of flow of hydrogen is from 0.1 to 2 liters per minute per kilogram, the carbon content of the iron being reduced to less than .005%, and 10 cooling the decarbonized iron in hydrogen to a temperature of below 500° C.

6. In the process of heat treating silicon iron, in combination, coating the silicon iron with a refractory permeable to hydrogen and carbon monoxide, oxidizing the coated iron to deposit 20 from .05% to .3% by weight oxygen over the surface of the steel, heating the silicon iron at a temperature of from 900° C. to 1300° C. in hy-

drogen for from 10 to 30 hours, the hydrogen having a dew point of -60° C. to 0° C., controlling the rate of flow of the hydrogen during the heating at from .1 to 1 liter per minute per kilogram, the heating reducing the carbon content of the iron to less than .005%, and cooling the decarbonized iron in hydrogen to a temperature of below 500° C. 5

7. In the process of heat treating silicon iron, in combination, coating the silicon iron with a 10 refractory, oxidizing the coated strip to deposit from .05% to .3% by weight oxygen over the surface of the iron, heating the silicon iron at a temperature of from 900° C. to 1100° C. in hydrogen or from 10 to 30 hours, the hydrogen having a dew point of from -60° C. to 0° C. when the rate of flow of hydrogen is less than 2 liters per minute per kilogram, the oxygen on the surface of the iron reacting with the carbon of the iron to reduce the carbon content to less than .005%, 15 and cooling the decarbonized iron in hydrogen to a temperature of less than 500° C. 20

TRYGVE D. YENSEN.