

June 20, 1972

KO KUMAI ET AL

3,671,337

PROCESS FOR PRODUCING GRAIN ORIENTED ELECTROMAGNETIC
STEEL SHEETS HAVING EXCELLENT MAGNETIC
CHARACTERISTICS

Filed Feb. 5, 1970

5 Sheets-Sheet 1

FIG.1

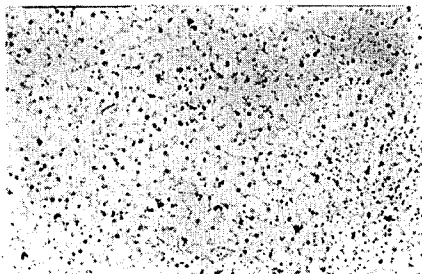
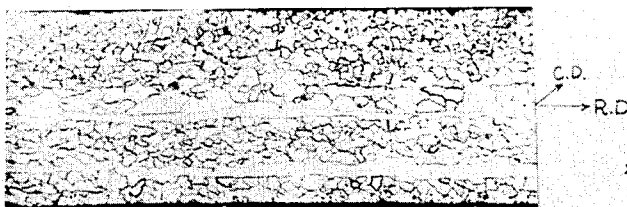


FIG.2



FIG.4



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FIG. 3

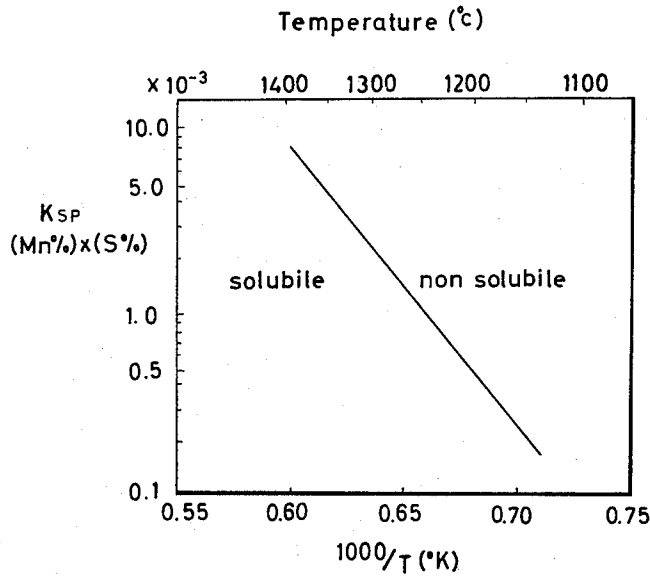


FIG. 7

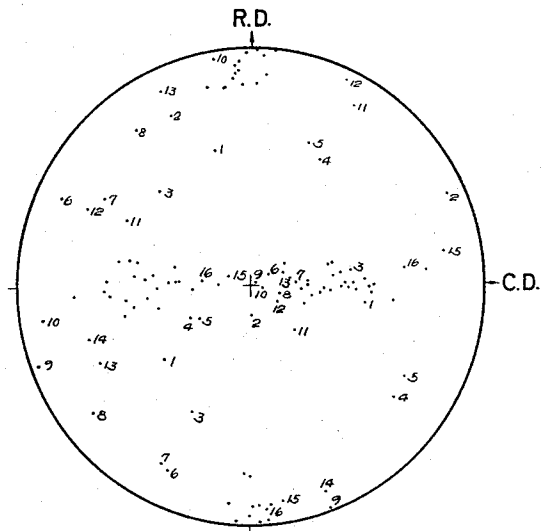
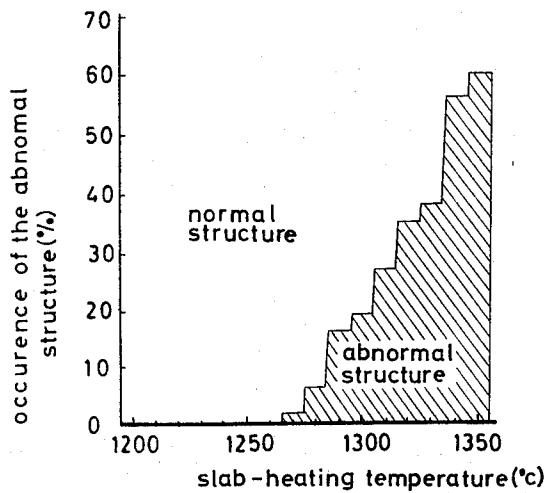


FIG. 8



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FIG.5

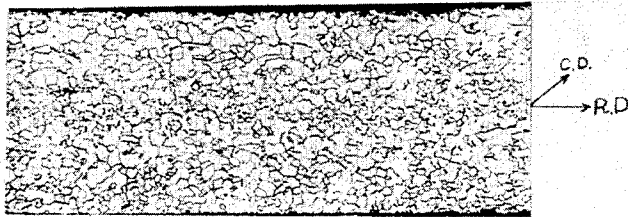


FIG.6

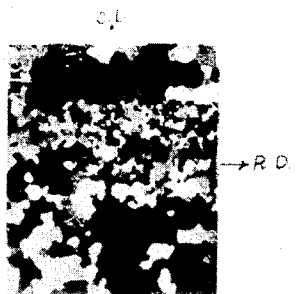
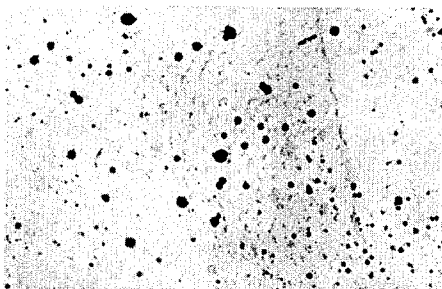


FIG.9



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FIG. 10

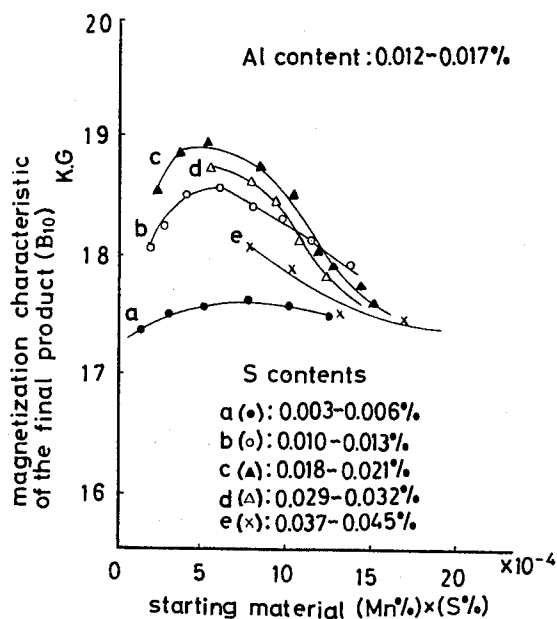
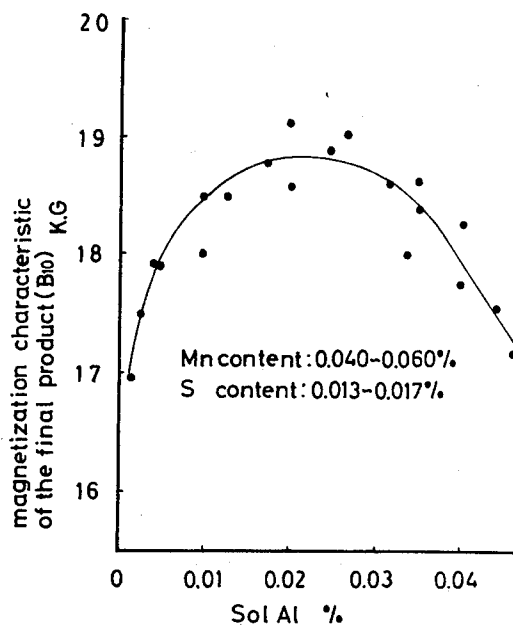


FIG. 11



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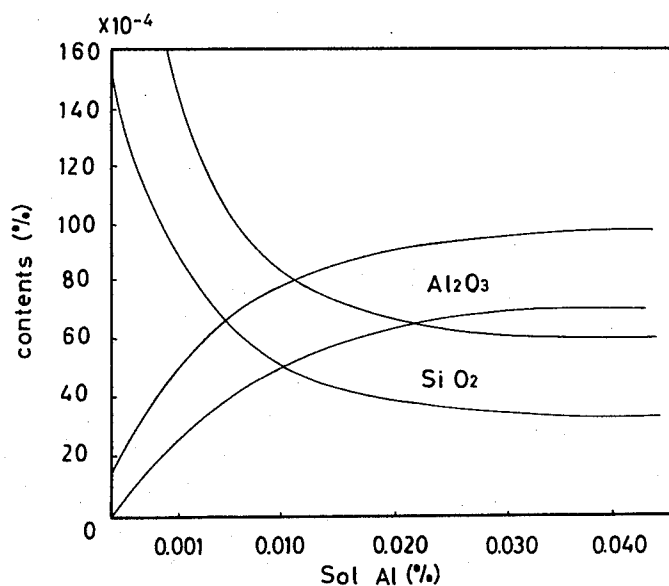
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FIG. 12



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PROCESS FOR PRODUCING GRAIN ORIENTED ELECTROMAGNETIC STEEL SHEETS HAVING EXCELLENT MAGNETIC CHARACTERISTICS

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Claims priority, application Japan, Feb. 21, 1969,

44/13,074

Int. Cl. H01f 1/16

U.S. Cl. 148—111

8 Claims

ABSTRACT OF THE DISCLOSURE

A process for producing grain oriented electromagnetic steel sheets having excellent magnetic characteristics. The steel contains MnS within a critical range and the Si content of said steel is controlled by the inclusion therein of Al as a deoxidant. The process comprises slab-heating a slab of the above-described steel at about 1150° C. to about 1280° C. and then hot rolling the steel followed by a two-step cold-rolling operation with an intermediate annealing step.

SUMMARY

This invention relates to a process for producing grain oriented electromagnetic steel sheets and strips containing not more than 4% by weight of silicon.

It is generally well-known that silicon steel consists of crystal grains having a body centered cubic lattice and that the direction of three mutually vertical edges $\langle 100 \rangle$ of this body centered cubic lattice is the direction of easiest magnetization. In grain oriented rolled electromagnetic sheet the $\langle 100 \rangle$ axis, i.e., the direction of easy magnetization, is disposed parallel to the direction of rolling and the $\{110\}$ plane is disposed parallel to the rolling plane. Crystallographically, this orientation is expressed as $\{110\} \langle 100 \rangle$ by the Miller index.

Thus, in grain oriented rolled electromagnetic steel sheet, crystal grains constituting the steel have a specific orientation. The formation of such crystal grains can be realized by the so-called secondary recrystallization phenomenon, namely by the selective growth of crystal grains have the $\{110\} \langle 100 \rangle$ orientation among the primary recrystallized grains during the final annealing stage of the steel sheet after the final sheet thickness is obtained by cold rolling.

Since grain oriented electromagnetic steel sheet is used as a soft magnetic material primarily for the iron cores of transformers and generators, it is important, as regards the magnetic properties, to have an eminent magnetization characteristic, i.e., the relation between the magnetic field intensity and the density of the magnetic flux, as well as a good iron loss characteristic, i.e., the relation between the magnetic flux density and the iron loss.

The quality of magnetization characteristic, generally expressed as B_{10} , depends on the density of the magnetic flux induced in the iron core by a known magnetic field. An iron core with a high B_{10} value is obtained from grain oriented rolled electromagnetic steel sheet containing large numbers of grains having the easily magnetized $\langle 100 \rangle$ axis along the direction of rolling.

Iron loss, generally expressed as W 15/50, is the energy

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loss when a known alternating magnetic flux density is applied to the iron core. It is well-known that, while the W 15/50 value is partially influenced by impurities, the specific resistance and the residual stress, the value is determined mainly by the magnetization characteristic of the grain oriented electromagnetic steel sheet comprising the iron core.

Thus, improving the magnetization characteristic (B_{10}) of the grain oriented electromagnetic steel sheet not only causes reduction of iron loss, but it also makes possible the reduction of the size of various electrical instruments, since only a relatively weak magnetic field is required to produce the desired magnetic flux density.

An object of this invention is to provide an economical industrial method for producing grain oriented rolled electromagnetic steel sheets and strips which have an excellent magnetization characteristic along the direction of rolling compared to the grain oriented electromagnetic steel sheets hitherto obtained and which have a B_{10} value of at least about 18.0 kilogausses (kg.).

The foregoing object is attained by subjecting steel containing less than about 4.0 weight percent Si, about 0.005 to about 0.040 weight percent acid-soluble Al, about 0.01 to about 0.03 weight percent S, and Mn in an amount such that the weight percent of Mn multiplied by the weight percent of S is about 0.0002 to about 0.0011 weight percent to the steps:

- (a) slab-heating a slab of said steel at about 1150° C. to about 1280° C.;
- (b) hot-rolling said steel slab; and
- (c) cold-rolling said steel by a two-step procedure with an intermediate annealing step.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an electronmicrograph showing a precipitate of MnS in hot rolled steel.

FIG. 2 is an electronmicrograph showing a precipitate of MnS in hot rolled steel.

FIG. 3 is a plot of the solubility of MnS in steel versus the slab-heating temperature.

FIG. 4 is an electronmicrograph showing abnormal primary recrystallized structure.

FIG. 5 is an electronmicrograph showing normal primary recrystallized structure.

FIG. 6 shows the macro etch structure of the abnormal structure of FIG. 4.

FIG. 7 shows the crystal orientations of each crystal shown in FIG. 6.

FIG. 8 is a graph showing the relationship between slab-heating temperature and the occurrence of abnormal primary recrystallized structure.

FIG. 9 is an electronmicrograph showing non-uniform MnS precipitate in hot-rolled steel strip.

FIG. 10 is a plot of magnetization characteristic versus the product of weight percent Mn $\times$ weight percent S in the starting material.

FIG. 11 is a plot of magnetization characteristic versus the weight percent of acid-soluble Al in the starting material.

FIG. 12 is a plot of the weight percent of SiO_2 and of Al_2O_3 versus the weight percent of acid-soluble Al in the steel.

DETAILED DESCRIPTION OF THE INVENTION

The steel slab used in the practice of this invention is obtained by deoxidizing the molten steel manufactured in the steel making furnace with sufficient aluminum to leave about 0.005 to about 0.040 weight percent of acid-soluble aluminum. Other constituents of the steel slab, expressed as weight percent of the over-all steel composition, are:

Si: less than about 4.0 weight percent,

C: about 0.06 weight percent,

N: about 0.004 to about 0.007 weight percent,

S: about 0.01 to about 0.03 weight percent,

Mn: sufficient that weight percent Mn $\times$ weight percent S is about 0.0002 to about 0.0011 weight percent.

The carbon content is not necessarily restricted to about 0.06 weight percent of the steel composition, but if the carbon content exceeds this amount, much time is required in a subsequent decarburization step and, accordingly, the economics of the process suffer.

In the process of this invention, the steel slab having the foregoing composition is slab-heated at about 1150° to about 1280° C., and preferably at about 1150° to less than about 1260° C.; hot-rolled to give hot-rolled steel strip; and the strip is then subjected to a two-step cold-rolling procedure with an intermediate annealing step. It is thus an object of this invention to obtain, by the above process, grain oriented rolled electromagnetic steel sheets with a B_{10} value not less than about 18.0 kg. along the direction of rolling after the final annealing step.

Such grain oriented rolled electromagnetic steel sheets having excellent magnetization characteristics such as a B_{10} value of not less than 18.0 kg. are obtained for the first time due to the increase in the number of crystal grains having the $\{110\}<100>$ orientation by the method of controlling the so-called secondary recrystallization phenomenon, namely by the selective growth of crystal grains having the $\{110\}<100>$ orientation among the primary recrystallized crystal grains during the final annealing stage.

The present inventors have ascertained that the following two factors are important for this purpose:

(1) Forming a dispersed phase precipitate suitable for secondary recrystallization; and

(2) Homogenizing the primary recrystallized structure prior to the secondary recrystallization.

In order to promote the secondary recrystallization, it is necessary to first form a dispersed phase precipitate. It is well-known that such a dispersed phase precipitate acts as a controlling agent for the grain growth of other crystal grains whereby the primary recrystallized grains having the $\{110\}<100>$ orientation grow selectively in the final annealing stage.

As for the dispersed phase precipitate, many impurities such as MnS, AlN, VN, TiC and the like are known. However, MnS has generally been adopted from among these impurities due to the fact that the amount of MnS contamination usually found as an impurity in steel is usually sufficient to promote secondary recrystallization without the necessity for adding additional MnS for this purpose.

In this invention, MnS is used chiefly as an impurity to form a dispersed phase precipitate. However, it is one of the main characteristics of this invention to give the MnS a suitable property as a dispersed phase precipitate whereby a uniformly dispersed, fine precipitate of MnS is obtained.

Industrially, it is economical to form a dispersed phase precipitate of MnS which is effective for secondary recrystallization at the hot-rolling stage, since the form change of MnS (solid solution formation, precipitation) occurs only at relatively high temperatures, i.e., above about 900° C. to about 1000° C.

Generally, the hot-rolling process consists of two

stages, namely, the slab-heating step, in which the slab obtained by the blooming or by the continuous casting is reheated, and the hot-rolling stage, in which the slab which has been reheated is hot-rolled to form a steel strip.

MnS precipitate in the slab before reheating in the slab-heating process has grown to a size greater than about 10 microns in diameter, and such a dispersed state of the precipitate is not effective for secondary recrystallization. In order to change the form of the large size MnS to obtain a precipitate with an effective dispersed state, it is first necessary to dissolve this large size MnS in solid steel in the slab-heating stage and then to precipitate the MnS in a fine state in the hot-rolling stage. Now it has been found that the dispersed state of MnS precipitate is greatly influenced by the formation of a solid solution of MnS in steel. Electronmicrographs showing the dispersed state of MnS precipitate in hot-rolled steel strip are given in FIG. 1 and FIG. 2. FIG. 1 shows a precipitate of MnS that had been sufficiently dissolved in the solid state in the slab-heating stage, and FIG. 2 shows an MnS precipitate resulting from insufficient solution of MnS in the solid state in the slab-heating stage.

The present inventors studied the effect of temperature on the formation of solid solutions of MnS in steel during the slab-heating stage, and obtained the relationship between the heating temperature and the solubility of MnS as shown in FIG. 3. From FIG. 3, it is obvious that the amount of MnS capable of forming a solid solution increases with an increase in the slab-heating temperature. It is concluded, therefore, that the formation of a solid solution of MnS, which is necessary to obtain an MnS precipitate with an effective dispersed state for the secondary recrystallization, becomes more complete by raising the slab-heating temperature. It is desirable, therefore, that the slab-heating temperature be sufficiently high.

While it was desirable, as mentioned above, that the slab-heating temperature be sufficiently high to form an MnS precipitate with the effective dispersed state for secondary recrystallization, it was found, on the other hand, that the slab-heating temperature has an influence on the primary recrystallized structure present prior to the secondary recrystallization, and the uniformity of the primary recrystallized structure is adversely affected by higher temperatures.

It was found during the course of experiments to obtain the relationship between the dispersed state of the MnS precipitate and the behavior of the secondary recrystallization that the dispersed state of the MnS precipitate in the hot-rolled steel sheet obtained under conditions of slab-heating temperatures sufficiently high, i.e., for example, 1320° C., to sufficiently dissolve MnS in a solid solution, was excellent as shown in FIG. 1, but the secondary recrystallization was locally insufficient in some parts of the final product. Accordingly, the magnetic characteristics of the steel strip in the final product were sometimes lacking in uniformity. After studying such materials lacking uniformity in magnetic characteristics as mentioned above, it was learned that in such cases the primary recrystallized structure at the final annealing consisted of both the abnormal structure shown in FIG. 4 and the normal structure shown in FIG. 5.

In the abnormal structure shown in FIG. 4 there exist elongated grains along the direction of rolling, and accordingly the uniformity of the primary recrystallized structure was injured. These elongated grains had a crystal orientation $\{110\}$ axis parallel with the direction of rolling and thereby prevented the growth of crystal grains having the $\{110\}<100>$ orientation in the secondary recrystallization stage of the final annealing where the crystal grains having the $\{110\}<100>$ orientation grow selectively. These elongated grains later grew

normally, but the overall rate of formation of crystal grains having the $\{110\} <100>$ orientation diminished.

FIG. 6 shows the macro etch structure of a part including the abnormal structure in FIG. 4 after the secondary recrystallization, and FIG. 7 is the (200) pole figure showing the crystal orientations of each of the crystal grains found in the macro etch structure in FIG. 6. It is clearly seen from the figures that the crystal orientations of the fine particles in FIG. 6 (numerals are given for the poles in FIG. 7) deviate markedly from the $\{110\} <100>$ orientation. As a consequence of the reduction in the number of crystal grains having the $\{110\} <100>$ orientation, the magnetization characteristic of the final product grew worse, in some parts of the product being below 18.0 kg., and in some instances, being even below 17.0 kg.

As mentioned above, it is clear that the secondary recrystallization is influenced not only by the dispersed state of the MnS precipitate, but also by the uniformity of the primary recrystallized structure present prior to the secondary recrystallization. Therefore, it is necessary to prevent the occurrence of the abnormal structure in the primary recrystallized structure as illustrated in FIG. 4. The present inventors have ascertained that the abnormal structure is influenced by the slab-heating temperature and that its occurrence can be completely prevented by controlling the slab-heating temperature so as not to exceed 1280° C., and preferably to keep the slab-heating temperature below 1260° C. FIG. 8 shows the relationship between the slab-heating temperature and the occurrence of the abnormal primary recrystallized structure. The samples were prepared as follows:

Steel slabs containing about 0.025% C. and about 3.1% Si were hot-rolled after slab-heating at various temperatures in the range between 1200° C. and 1350° C., and were then cold-rolled by the conventional two-step cold-rolling procedure including an intermediate annealing step for the occurrence of recrystallization, and the steel strips thus obtained were subjected to the secondary recrystallization treatment. One hundred points were examined on each sample and a plot of slab-heating temperature versus the occurrence of abnormal structure was made. It is obvious from this graph (FIG. 8) that the primary recrystallized structure is uniform when the slab-heating temperature is selected so as not to exceed 1280° C. and preferably to be below 1260° C.

However, when the slab-heating temperature is too low, i.e., below 1150° C., a satisfactory dispersed phase precipitate is not obtained due to the extremely small amount of MnS in solid solution. Thus, the object of this invention, to obtain excellent magnetization characteristics, cannot be achieved. Moreover, as a consequence, the finishing temperature in the subsequent hot-rolling step is too low, and the shape of the hot-rolled steel strip is unsatisfactory resulting in poor yields. With these points in mind, it becomes apparent that the range of the slab-heating temperature necessary to prevent the occurrence of the abnormal primary recrystallized structure is 1150° to about 1280° C., and preferably 1150° C. to below about 1260° C.

In order to obtain a uniformly dispersed precipitate of MnS under the slab-heating temperatures mentioned above, the amount of MnS to be added to the steel should be in agreement with the amount of MnS capable of forming a solid solution in the abovementioned temperature range. From FIG. 3, it is apparent that the amount of MnS capable of forming a solid solution in steel in the slab-heating temperature range between 1150° to below 1260° C. can be expressed by the relation:

$$[\text{wt. percent Mn}] \times [\text{wt. percent S}] = 0.0002-0.0011 \text{ wt. percent}$$

Since the amount of Mn contained in the dispersed phase precipitate is undefined from this expression alone, the range of the amount of sulfur must be decided first. Accordingly, a feature of this invention is to define the

amount of S in the silicon steel slab used in this invention as:

$$S=0.010-0.030 \text{ wt. percent}$$

and to define the amount of Mn as corresponding to the expression:

$$[\text{wt. percent Mn}] \times [\text{wt. percent S}] = 0.0002-0.0011 \text{ wt. percent}$$

By defining the amounts of Mn and S, and by controlling the slab-heating temperature as mentioned above, sufficient MnS can be dissolved in solid steel in the slab-heating stage to prevent the occurrence of abnormal structure in the primary recrystallized structure. However, in some instances even with these precautions, the secondary recrystallization phenomenon does not occur to a sufficient degree, and consequently a degradation in the magnetization characteristics is observed. Since it was found that such a degradation of magnetization characteristics frequently occurred when the initial steel composition contained large quantities of silicious inclusions, the influence of silicious inclusions was examined. As a result, it was ascertained that silicious inclusions could combine readily with MnS, and accordingly the formation of a solid solution of MnS was hindered in the slab-heating stage. Moreover, the dispersed phase precipitate of MnS became non-uniform in the subsequent precipitation stage because the silicious inclusions induced the precipitation of MnS. Finally, the quantity of MnS which is effective for the secondary recrystallization is diminished.

FIG. 9 is an electromicrograph a non-uniform precipitate of MnS in a hot-rolled steel strip. In this case, the amount of silicious inclusion was abundant and, while MnS was sufficiently dissolved during the slab-heating stage, the MnS precipitate combined with the silicious inclusion in the subsequent precipitation stage, and consequently the dispersed state of the MnS precipitate was not uniform.

In order to reduce silicious inclusions and to obtain a uniformly dispersed precipitate of MnS, deoxidation by the use of Al, Ti and the like can be carried out without consuming Si. The present inventors have found that satisfactory results can be obtained by using Al as a deoxidant and in such quantities that more than about 0.005 weight percent acid-soluble Al remains after deoxidation.

The relationship between the amount of acid-soluble Al and the amounts of SiO_2 and Al_2O_3 in molten steel is shown in FIG. 12. As seen in FIG. 12, by using Al as a deoxidant the amount of silicious inclusion in the molten steel can be reduced and the MnS precipitate is uniformly dispersed. The time for the addition of Al in order to bring about the deoxidation by Al and to reduce silicious inclusion is optional between the tapping and the teeming of the molten steel. Since the order of the addition of Al and Si has no effect on the amount of silicious inclusion, Al can be added at any time.

Thus it is a feature of this invention to bring about deoxidation by Al so as to leave in the steel acid-soluble Al in the range between about 0.005 to about 0.040 weight percent. In such a case, acid-soluble Al reacts with nitrogen to form AlN. Since MnS co-exists with AlN, when the amount of MnS in the dispersed phase precipitate is insufficient, it is supplemented with AlN; and when the amount of MnS is proper, a better dispersed state of the precipitate can be obtained through the effect of the AlN. Consequently, the secondary recrystallization phenomenon is more stabilized. In this way, by using Al as a deoxidant, very desirable results can be obtained. Thus, the use of Al as a deoxidant has the effect of reducing the amount of silicious inclusion, improving the dispersed state of the MnS precipitate, and AlN which is formed co-exists in the dispersed phase precipitate. Consequently, the magnetization characteristic is improved remarkably.

The above improvement can readily be understood from FIG. 10 and FIG. 11 which show the effects of the amounts of S, Mn and acid-soluble Al in the initial steel composition on the magnetization characteristic along the direction of rolling of the final product after the secondary recrystallization. The samples for the test were prepared as follows: small steel ingots with a weight of 100 kg., containing about 0.030 weight percent C, about 3.1 weight percent Si and varying amounts of S, Mn, and Al were prepared by induction melting and casting in open air whereby the nitrogen content became 0.005–0.007 weight percent. The ingots were forged while hot to 18 mm. x 150 mm. slabs. After being heated to 1240° C., the slabs were hot-rolled to obtain 2.5 mm. hot-rolled sheets. The sheets thus obtained were rolled by the two-step cold-rolling method, with an intermediate annealing step for recrystallization to occur, to a final gauge of 0.35 mm. The resultant sheets were decarburized and annealed at 1150° C. for three hours to obtain the final products.

While the object of this invention can be accomplished when the amount of acid-soluble Al is more than 0.005 weight percent, taking into consideration the increase of iron loss due to the formation of aluminous inclusions by oxidation of aluminum in the later stages, the upper limit of Al as acid-soluble Al is selected so as not to exceed 0.040 weight percent.

The principle of this invention, to stabilize the secondary recrystallization by improving the dispersed state of the MnS precipitate and, at the same time, having AlN co-existent in the dispersed state precipitate, is obtained by the combination of the following steps, and is quite novel. These steps are to prevent the occurrence of abnormal structure in the primary recrystallized structure by defining the upper limit of the slab-heating temperature to be 1280° C. and preferably less than 1260° C., to dissolve MnS to a sufficient degree in solid steel below the above-mentioned slab-heating temperature, by defining the amounts of Mn and S that are present, and also by intensifying deoxidation by Al to diminish silicious inclusion.

In the practice of this invention, the conventional two-step cold-rolling procedure including the intermediate annealing step for recrystallization is adopted for the treatment of the hot-rolled sheet to thereby convert said hot-rolled sheet to the final product. While the hot rolled sheet obtained in the intermediate stage of this invention gives a stable secondary recrystallization phenomenon in the final annealing step without the need of special treatment in the following stage, the two-step cold-rolling procedure is adopted in order to stabilize more satisfactorily the magnetization characteristics of the product.

It is disclosed in U.S. Pat. 3,159,511 that by using a steel slab containing 0.01–0.04% of acid-soluble Al and by employing as slab-heating temperature below 1260° C., a relatively good magnetization characteristic can be obtained even when a one-step cold-rolling procedure is used. The dispersed phase precipitate for the secondary recrystallization, however, differs from that of the present invention in that AlN alone is used. It is apparent that there exist fundamental differences between the invention of U.S. Pat. 3,159,511 and that of the present invention in respects other than the slab-heating temperature. In the instant invention, an excellent magnetization characteristic is obtained by the following process: MnS is used as the primary dispersed phase precipitate, and Al is used as the deoxidant in order to reduce the amount of silicious inclusion. As a result, the dispersed state of the MnS precipitate is improved and, in addition, AlN co-exists as a dispersed phase precipitate. Also, a two-step cold-rolling procedure is used in the instant process. The present inventors have found that a satisfactory magnetization characteristic cannot be obtained in their process by the use of AlN alone as the dispersed phase precipitate. This is illustrated in curve *a* of FIG. 10.

A process employing a temperature range between

1260° C. and 1399° C. for the slab-heating temperature is disclosed in Japanese Pat. No. 216,505 to M. F. Littmann. To the contrary, in the present invention the upper limit of the slab-heating temperature is restricted to 1280° C. and is preferably below 1260° C. in order to prevent the occurrence of abnormal structure in the primary recrystallized structure. Furthermore, the lower limit of the slab-heating temperature of the instant invention, i.e., 1150° C., is far below that of said Japanese patent. To carry out slab-heating at the high temperatures disclosed in the Littmann patent causes degradation of the magnetization characteristic due to the formation of abnormal structure, and, furthermore, it is economically disadvantageous for the following reasons:

(1) There is a decrease in yield due to the formation of a mixed oxide between iron and silicon at the slab surface,

(2) There is a decrease in yield due to the occurrence of a flaw on the backside of the slab formed by the mixed oxide deposited on the hearth of the slab-heating furnace,

(3) There is a decrease in operating efficiency due to shutdown time necessary to remove the deposit on the hearth of the slab-heating furnace,

(4) There is a damage to the hearth bricks in removing the deposit on the hearth of the slab-heating furnace and there is a decrease in the life of the heating furnace due to damage to the refractories caused by the high operating temperature of the furnace, and

(5) There is an increase in the cost of fuel for maintaining the furnace and the slab at the higher temperatures.

In U.S. Pat. 2,826,520 to R. L. Rickett, a process is disclosed in which a starting material containing less than 0.025% C, less than 0.08% Mn, less than 0.005% P, less than 0.025% S, and less than 0.15% Cu is slab-heated between 1218° C. and 1245° C. and hot-rolled at a finishing temperature of above 945° C. Compared to the present invention, however, a pronounced difference lies in the action of Al. Whereas Al acts to improve the dispersed state of the MnS precipitate by diminishing the amount of silicious inclusion and also acts to form a dispersed phase precipitate by coexisting as AlN with the MnS in the instant invention, Al is not used in the invention of Rickett. Consequently, the magnetization characteristic of Rickett, as expressed by the magnetic permeability at a magnetic flux density at 15 kg. is only 18,121 gauss. On the other hand, the magnetic permeability obtained in the instant invention is at least over 20,000 gauss.

The following examples will tend to more fully illustrate the invention.

Example 1

To molten steel smelted in a basic converter were added 1.4 kg./ton of Al and silicon steel slabs were bloomed. The steel slabs contained 0.016% S, 0.054% Mn, 0.025% C, 3.09% Si, 0.021% acid-soluble Al and 0.0058% N, the foregoing all being weight percents. One slab was slab-heated at 1240° C. for two hours and the other was slab-heated at 1360° C. for two hours. Both slabs were then hot-rolled to obtain 2.5 mm. hot-rolled steel strips. These strips were rolled by the two-step cold-rolling method with an intermediate annealing step at 850° C. for three minutes to a final gauge of 0.35 mm. (the rate of reduction in the secondary cold-rolling step being 55%), decarburized at 800° C. for three minutes in an atmosphere of wet hydrogen and finally annealed at 1170° C. for 20 hours in an atmosphere of hydrogen.

The magnetic characteristics of each of these samples were determined at 100 points along the rolling direction. The results were as follows:

Slab-heating: 1240° C. for two hours

B₁₀: 18.1–18.4 kg.

W 15/50: 1.02–1.05 watts/kg.

Slab-heating: 1360° C. for two hours

B₁₀: 17.4–18.4 kg.

W 15/50: 1.02–1.16 watts/kg.

The distributions of magnetization characteristics along the longitudinal directions of the samples were as follows:

	Slab heating	
	1,240° C., 2 hours	1,360° C., 2 hours
B ₁₀ , kg.:		
18.4-----	14	17
18.3-----	42	25
18.2-----	36	24
18.1-----	8	13
18.0-----	0	4
17.9-----	0	9
17.8-----	0	1
17.7-----	0	5
17.6-----	0	0
17.5-----	0	2

The foregoing shows the superior magnetization characteristics obtained when the steel is slab-heated at a temperature below 1280° C., i.e., 1240° C., according to the procedure of the instant invention.

Example 2

To molten steel smelted in a basic converter were added 1.4 kg./ton of Al and a silicon slab was bloomed. The steel slab contained 0.022% S, 0.045% Mn, 0.040% C, 3.04% Si, 0.032% acid-soluble Al, and 0.0065% N, the foregoing all being weight percents. The slab was slab-heated at 1250° C. for two hours and was then hot-rolled to yield a 2.5 mm. hot-rolled steel strip. The strip was rolled by the two-step cold-rolling procedure with an intermediate annealing step at 850° C. for three minutes to a final gauge of 0.35 mm. (the rate of reduction in the secondary cold-rolling being 55%), decarburized at 800° C. for three minutes in an atmosphere of wet hydrogen, and finally annealed at 1170° C. for 20 hours in an atmosphere of hydrogen.

The magnetic characteristics along the direction of rolling of the steel strip obtained were:

B₁₀: 18.1–18.3 kg.
W 15/50: 1.03–1.06 watts/kg.

The deviation of magnetic characteristics along the strip was only slight.

Example 3

To molten steel smelted in a basic converter was added Al in the amount 1.2 kg./ton of steel and the silicon steel slab was bloomed. The composition of the slab was controlled to contain 0.014 weight percent S and 0.063 weight percent Mn. Other minor constituents were 0.048 weight percent C, 3.11 weight percent Si, 0.014 weight percent acid-soluble Al, and 0.0053 weight percent N. The slab was slab-heated at 1230° C. for three hours and then hot-rolled to yield a 2.5 mm. hot-rolled steel strip. The strip was rolled by the two-step cold-rolling method with intermediate annealing at 850° C. for three minutes to a final gauge of 0.35 mm. The rate of reduction in the secondary cold-rolling procedure was 55 percent. The steel strip was decarburized at 800° C. for three minutes in an atmosphere of wet hydrogen, and finally annealed at 1170° C. for 20 hours in a hydrogen atmosphere.

The magnetic characteristics along the direction of rolling of the product obtained were:

B₁₀: 18.4–18.7 kg.
W 15/50: 0.97–1.01 watts/kg.

and the deviation of magnetic characteristics in the product was only slight.

Example 4

To molten steel smelted in a basic converter was added Al in the amount 1.3 kg./ton of steel and silicon steel slabs were bloomed. The composition of the slabs was adjusted so as to contain 0.012 weight percent S, 0.030 weight percent Mn, 0.034 weight percent C, 3.20 weight percent Si, 0.015 weight percent acid-soluble Al, and 0.0055 weight percent N. One of the slabs was slab-heated at 1170° C. for two hours and the other was slab-heated at 1200° C. for two hours. Both slabs were hot-rolled to obtain 2.5 mm. hot-rolled steel strips. These strips were rolled by the two-step cold-rolling procedure with an intermediate annealing step at 850° C. for three minutes to a final gauge of 0.35 mm. The rate of reduction in the secondary cold-rolling was 55%. The steel strips were decarburized at 800° C. for three minutes in an atmosphere of wet hydrogen and finally annealed at 1170° C. for 20 hours in an atmosphere of hydrogen.

The magnetic characteristics of these products along the direction of rolling were:

Slab-heating at 1170° C. for two hours:

B₁₀: 18.0–18.3 kg.
W 15/50: 1.00–1.05 watts/kg.

Slab-heating at 1200° C. for two hours:

B₁₀: 18.1–18.4 kg.
W 15/50: 0.98–1.02 watts/kg.

The deviations of the magnetic characteristics in the samples were only slight.

Example 5

The following experiment illustrates that a product with satisfactory magnetic characteristics cannot be obtained from silicon steel without using Al as the deoxidant even when the slab-heating treatment is carried out in the temperature range of this invention. Molten steel obtained by the smelting in a basic converter was bloomed without deoxidation by Al to a silicon steel slab containing 0.025 weight percent C, 3.15 weight percent Si, 0.050 weight percent Mn, 0.017 weight percent S, 0.0065 weight percent N, and a trace of acid-soluble Al. The slab was slab-heated at 1240° C. for two hours and hot-rolled to yield a 2.5 mm. hot-rolled steel strip. The strip was rolled by the two-step cold-rolling procedure with an intermediate annealing step at 850° C. for three minutes to a final gauge of 0.35 mm. The rate of reduction in the secondary cold-rolling was 55%. The strip was decarburized at 800° C. for three minutes in an atmosphere of wet hydrogen and finally annealed at 1170° C. for 20 hours in a hydrogen atmosphere. The magnetic characteristics of the product were:

B₁₀: 1.65–1.72 kg.
W 15/50: 1.16–1.37 watts/kg.

In this example, since deoxidation by the use of Al was not carried out, there was a high level of silicious inclusion. Consequently, the dispersed phase precipitate of MnS in the secondary recrystallization was not uniform, and as a result the magnetic characteristics were degraded. The deviations of the magnetic characteristics in the product were high.

As described above, this invention is concerned with a profitable industrial manufacturing method for grain oriented electromagnetic steel having excellent magnetization characteristics, with a B₁₀ value of at least 18.0 kg. The process consists of deoxidizing molten steel smelted in a steel-making furnace with Al in a quantity such that the steel contains about 0.005–0.04 weight percent of acid-soluble Al. The composition of the slab is adjusted to contain about 0.010–0.03 weight percent S and an amount of Mn such that the product [weight percent Mn] × [weight percent S] = 0.0002–0.0011 weight percent of the steel composition. Other constituents of the steel are less than 0.06 weight percent C and less than 4.0 weight percent Si. The slab is slab-heated at a temperature between about 1150° and 1280° C., and preferably between about 1150° and 1260° C. The slab is then hot-rolled followed by a two-step cold-rolling procedure. This invention affords, moreover, great advantages in manufacturing the product; for instance, due to the diminution of the damage to the slab-heating furnace, not only is the life of the slab-heating furnace prolonged, but also the furnace operation is quite convenient.

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What is claimed is:

1. In a process for producing a single-oriented electromagnetic steel sheet and strip comprising hot rolling slab into hot rolled sheet, subjecting the resulting hot rolled sheet to a two-step cold rolling procedure including an intermediate annealing step and subjecting the resulting cold rolled sheet to decarburizing and final annealing steps, the improvement which comprises that a silicon steel slab containing less than 4.0 weight percent Si, 0.005–0.040 weight percent acid-soluble Al, 0.01–0.03 weight percent S, and Mn in an amount such that the weight percent of Mn multiplied by the weight percent of S is 0.0002–0.0011 be heated to 1150°–1280° C. prior to being hot rolled.

2. The process of claim 1 wherein the slab-heating temperature is from about 1150° C. to less than about 1260° C.

3. The process of claim 1 wherein the intermediate annealing step comprises heating said steel at about 800°–900° C.

4. The process of claim 1 wherein the decarburizing step comprises heating the cold-rolled steel in wet hydrogen at about 750°–950° C.

5. The process of claim 1 wherein annealing the decarburized steel comprises heating said decarburized steel at about 1100°–1200° C. in a hydrogen atmosphere.

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6. The process of claim 1 wherein the intermediate annealing step comprises heating said steel at about 850° C. for about 3 minutes.

7. The process of claim 6 wherein the decarburizing step comprises heating the cold-rolled steel in wet hydrogen at about 800° C. for about 3 minutes.

8. The process of claim 7 wherein annealing the decarburized steel comprises heating said decarburized steel at about 1170° C. for about 20 hours in a hydrogen atmosphere.

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