Method for producing grain-oriented magnetic silicon steel.

Abstract: Method for producing grain-oriented magnetic silicon steel sheets having a high austenite content, preferably larger than 40%, during hot rolling, including the steps of preparing a melt of steel, hot rolling, high temperature annealing, cold rolling, decarburization and final annealing, wherein the melt of steel contains at least 0.1% by weight of manganese and/or at least 0.2% by weight of copper and wherein aluminium nitride (A1N) is used as the main inhibitor.
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Method for producing grain-oriented magnetic silicon steel

The present invention relates to a method for producing grain-oriented magnetic silicon steel sheets having a high austenite content, preferably larger than 40 %, during hot rolling, including the steps of preparing a melt of steel, hot rolling, high temperature annealing, cold rolling, decarburization and final annealing.

High performance transformers are an essential part of the modern energy infrastructure. With an increasing need for energy conservation and an increasing requirement for high performance transformers, the importance of a transformer core with a high permeability and a low core loss increases. One of the most successful materials in this regard is grain-oriented silicon steel. Grain-oriented silicon steel provides several important features for an improved transformer core performance. The addition of silicon reduces the conductivity of the steel, therefore reducing core loss due to eddy currents. The formation of oriented grains causes an increase in anisotropy of the material. Therefore the permeability of the material is also anisotropic. The permeability in one spacial direction is increased, the permeability in the other directions is decreased. Since only permeability in one direction is decisive for the performance of the transformer, improved transformers can be realised with this material.

Another important source of core loss is the presence of hysteresis of the magnetization of the transformer core. Hysteresis can be caused by impurities. Therefore the formation of those in the silicon steel has to be avoided.

Most commercial grain oriented steels with high permeability use aluminium nitride as an inhibitor during hot rolling. Here, it is of considerable importance to ensure fine precipitation of the inhibitor particles through all production
steps from steel making to hot rolled sheet annealing. It is well known that the solubility of aluminium nitride in the austenite phase is considerably higher than that in the ferrite phase, so it is essential to maintain a certain volume fraction of the austenite phase in order to first dissolve aluminium nitride at the soaking temperature and then precipitate fine aluminium nitride particles at the cooling stage of the hot rolled sheet annealing process.

It is common practice to increase the austenite fraction formed in the silicon steel by increasing the carbon content. However, high carbon contents lead to prolonged decarburization time, resulting in decreased productivity. Furthermore, low carbon levels are essential for achieving high drawability of the steel during rapid heating.

An object of the present invention is to provide a method for producing improved grain-oriented magnetic silicon steel with a high austenite fraction during hot rolling.

The object is achieved according to the present invention by a method, wherein the melt of steel contains at least 0.1% by weight of manganese and/or at least 0.2% by weight of copper and wherein aluminium nitride is used as the main inhibitor.

The present invention is motivated by the idea, that aluminium nitride should be used as the main inhibitor. However the high carbon contents necessary to ensure sufficient solubility and fine precipitation of the inhibitor particles should be avoided. Therefore another way has to be found to increase the fraction of the austenite phase in the steel during hot rolling. According to the present invention this is achieved by adding copper and manganese to the melt of steel without increasing C or decreasing Si contents. Both these metals can strongly increase the fraction of austenite in the steel during the hot rolling step. To give an example of this, the fraction of austenite in a steel that contains 2.9% by
weight of silicon increases by approximately 50% when the manganese content is varied between 0.1% by weight and 0.3% by weight. A similar increase is observed when increasing the content of copper from 0.2% by weight to 0.6% by weight.

Therefore it is possible to ensure sufficient austenite content at lower carbon contents. This provides several advantages. For one the decarburization time is shortened. This strongly increases productivity. Also low carbon content is essential for high drawability of the steel during rapid heating.

Preferably, the melt of steel contains at least 0.15% by weight of manganese and/or 0.3% by weight of copper. An increase in copper and/or manganese content can further increase the fraction of austenite during hot rolling. At the same time, if a certain minimum fraction of austenite is required, the carbon content can be lowered further. Till now there are no known negative side effects for the use as transformer material when increasing the manganese or copper content.

As previously described it is advantageous for the efficiency of the production and the drawability of the hot steel, if the carbon content is low. Therefore a melt of steel containing less than 0.07% by weight of carbon can be used in this process. The loss in austenite fraction by the reduction of the carbon content can be made up by adjusting the copper and/or manganese content. A further reduction of the carbon content below 0.04% by weight is also possible.

A further improvement of the described method can be achieved by keeping the content of sulphur contained in the melt of steel lower than 0.01% by weight. Sulphur might lead to the formation of manganese sulfide or copper sulfides. While both have their place in steel production, the formation is not desired here. The formation of manganese sulfide or copper sulfide might effectively reduce the amount of manganese or
copper in the alloy therefore reducing the fraction of the austenite phase during hot rolling. Also there is a chance of the formation of defects in the final steel leading to a higher probability of hysteretic behaviour.

As previously described, the addition of silicon to the melt of steel reduces the conductivity of the final product, therefore reducing core loss due to eddy currents. Therefore the melt of steel can contain 2.5 % to 6.6 % by weight of silicon. While it is generally desirable to reduce conductivity of the transformer core as far as possible, high concentrations of silicon can cause embrittlement during cold rolling and/or cutting. An increase in the silicon content can also reduce the fraction of austenite during hot rolling. Therefore typical values of 2.9 % to 3.4 % by weight of silicon can be used.

The melt of steel can contain up to 1.2 % by weight of chromium. The addition of chromium can help to reduce the levels of carbon while maintaining the desired ratio of ferrite to austenite during hot rolling. A chromium content below 1.2 % by weight promotes the formation of austenite, whereas chromium contents above 1.2 % by weight have adverse effects on decarburization and can lead to the formation of an isolating glass film.

In the production of high permeability grain-oriented silicon steels, the evolution of a sharp (110)<001> texture, also known as Goss texture, during secondary recrystallization is essential for achieving high magnetic properties. For the formation of this secondary recrystallization texture it is necessary to secure the formation of primary recrystallized Goss grains in the subsurface regions after decarburization. Aluminium nitride is used as main inhibitor and high reduction ratios are used. Therefore the method for production of steel sheets according to the present invention can contain at least one cold rolling step with a reduction of over 85 %.
To achieve a more uniform distribution of the inhibitor forming elements in the bulk slabs, the steel slabs can be heated to the pure ferrite region prior to hot rolling. Heating to very high temperatures can lead to an increase of energy consumption and unwanted liquid slack formation. Therefore a low temperature for the transition between the mixed austenite and ferrite phase region and the pure ferrite phase is desirable. Therefore at least one of the weight percentages of silicon, manganese and copper in the melt of steel can be varied, while keeping the carbon content constant, to achieve a given temperature of the phase transition from the mixed two-phase (austenite and ferrite) region to the pure ferrite phase.

It is advantageous to perform the variation by using a formula for the phase transition temperature of the shape:

\[ T_{ph} = a - b \cdot n(Si) + c \cdot n(Cu) - d \cdot n(Mn) + e \cdot n(Si) \cdot n(Mn) , \]

where \( n(Si) \), \( n(Cu) \) and \( n(Mn) \) are the concentrations in weight percent of silicon, copper and manganese, and \( a, b, c, d \) and \( e \) are constants determined by theory and/or experiment.

The use of a formula for determining the phase transition temperature has the advantage, that once parameters are established, a perfect combination of ingredients can be found simply by calculation. Other approaches require experimentation with different mixtures. Especially due to the high temperatures involved, this is an elaborate and expensive process.

In addition, the present invention relates to grain-oriented magnetic silicon steel produced by a method as described above.

The above and other aspects, features and other advantages of the present invention will be more clearly understood from
the following detailed description taken in conjunction with accompanying drawings, in which:

fig. 1 is a flow chart of a method for producing grain-oriented magnetic silicon steel sheets according to the present invention,

fig. 2 is a diagram showing the relative solubility of aluminium nitride in the ferrite and the austenite phase,

fig. 3 is a diagram showing the maximum austenite fraction in weight % depending on the silicon and manganese content,

fig. 4 is a diagram showing the maximum austenite fraction in weight % depending on the silicon and copper content, and

fig. 5 is a diagram showing the temperature of the phase transition from the mixed austenite and ferrite phase region to the pure ferrite phase depending on the copper and silicon content.

Fig. 1 is a flow chart of a method for producing grain-oriented magnetic silicon steel sheets.

As shown in fig. 1, the process may include the steps of steel making (S1), slab reheating (S2), hot rolling (S3), high temperature annealing (S4), heavy cold rolling (S5), decarburization (S6), final annealing (S7) and heat flattening and coating (S8).

In the steel making step (S1) the components of the future alloy are combined in a ladle and cast into slabs. The components of the alloy are small amounts of carbon, silicon, manganese, nitrogen, copper and aluminium, with iron making up the rest of the balance. In the exemplary embodiment 2.9 % of
silicon, 0.3 % of manganese, 0.4 % of copper and low concentrations of carbon (< 0.04 %) and sulphur (< 0.01 %) are used. The aluminium and nitrogen contents are adjusted such that full solubility of the aluminium nitride is still given.

Since the slabs start cooling down immediately after casting, a reheating step (S2) is performed. Here the slabs are heated up to the pure ferrite region. Since a rather low silicon content is used in the example the heating needs to be up to rather high temperatures of more than 1300 °C. The reheating ensures a more uniform distribution of the inhibitor forming elements, aluminium and nitrogen in the bulk of the slabs.

After the slab reheating step (S2) the hot rolling step (S3) is performed. For the production of grain-oriented steel with a high magnetic permeability it is essential that a high solubility of the aluminium nitride is achieved. In the relevant temperature range there is up to an order of magnitude difference in the solubility of aluminium nitride in the austenite and the ferrite phase. Therefore hot rolling is preferably to perform in the region of the phase diagram where both, the austenite and the ferrite phase are present. At 2.9 % silicon content and 0.3 % manganese content the maximum concentration of austenite by weight % (approx. 43 %) is expected at a bit less than 1200 °C. This temperature is maintained throughout the hot rolling process.

A high temperature needs to be maintained during the high temperature annealing phase (S4), due to the fact that silicon steel has to be cooled rapidly from the region where the austenite volume fraction exceeds a specified value.

After the prior steps, there is a fine precipitation of aluminium nitride present in the cold rolling step (S5). Heavy cold rolling with a high reduction, larger than 85 %, in the presence of aluminium nitride as a main inhibitor, leads to the formation of the primary recrystallisation texture after decarburization annealing (S6). The primary recrystallisation
texture is essential for the formation of a sharp Goss texture later in the process. A high carbon content could lead to the formation of additional defects in the steel sheets, therefore increasing the likelihood of hysteretic behaviour. To achieve a low core loss, this has to be avoided. Thanks to the low carbon content, which can be achieved due to the addition of manganese, decarburization is rather fast.

After decarburization (S6) a final annealing step (S7) ensures the proper formation of a sharp (110)<001> secondary recrystallisation texture. For a finished product, that can be used in a transformer, heat flattening and coating steps (S8) can be applied.

As another example, a similar melt of steel with a low manganese content (≤0.1 %) and a copper content of 0.6 % can be used. The further steps in the production process remain the same, since the changes in the temperatures for the maximum austenite content and the phase transition are minimal. The maximum fraction of the austenite phase is slightly lower in this case, approx. 38 %.

Obviously all melts of steel, that contain, copper and/or manganese, in concentrations in weight % of at least 0.1 % and 0.05 % respectively can also be used.

Fig. 2 shows a diagram, demonstrating the strongly different solubility of aluminium nitride in the austenite and ferrite phase (Takahashi et al., Journal of Magnetism and Magnetic Materials 160 (1996) 98 – 101). The ferrite phase, also known as a-iron is shown as the lower line, the austenite phase, also known as γ-iron is shown as the upper line. The concentration of aluminium nitride is given in logarithmic scale. The reciprocal of temperature is plotted on the x-axis to obtain a linear relationship. By comparing the values of the two lines in the relevant temperature range, easily achieved by referring to the temperature values shown on top of the plot, it is easily recognisable, that the solubility of $A_1N$
in the austenite phase is roughly one order of magnitude larger in the given range.

Fig. 3 shows a diagram collecting various data on the influence of the manganese and the silicon concentration on the maximum of the austenite fraction in weight %. The numbers shown for each data point in the plot represent the temperature, at which the austenite fraction reaches its maximum. Two general trends can be easily seen in this diagram. In general an increase of the silicon content at a constant manganese and carbon content reduces the fraction of the austenite phase. Therefore for higher silicon contents normally an increase in carbon content is necessary, leading to longer decarburization times. It is also seen, that at the same silicon content an increase in the manganese content increases the austenite fraction strongly. At the silicon content of 2.9 % by weight an increase of the manganese content from 0.1 % by weight to 0.3 % by weight increases the austenite fraction from approximately 30 % to nearly 45 %. Considering that normally 40 to 50 % by weight of austenite are required for a proper solution of aluminium nitride, the addition of manganese allows the production of grain-oriented steels with high magnetic permeability with a silicon content, that would otherwise require a higher carbon content.

Fig. 4 shows a diagram of the maximum weight fraction of austenite versus the silicon content and the copper content. Here, a similar behaviour is observed. An increase in the concentration of copper leads to an increase of the austenite fraction at all silicon contents. In general slightly more copper is necessary to achieve a similar effect, but the overall result is similar.

Fig. 5 shows a diagram of the phase transition temperature between the mixed ferrite austenite phase region and the pure ferrite phase dependant on the copper content and the silicon content. The symbols show the phase transition temperatures, while the lines represent fits, according to the expected de-
pendency of the transition temperature on the copper concentration. The general dependence of the transition temperature on the copper concentration, the manganese concentration and the silicon concentration at a fixed carbon concentration can be given as:

\[ T_{p_h} = a - b \cdot n(Si) + c \cdot n(Cu) - d \cdot n(Mn) + e \cdot n(Si) \cdot n(Mn), \]

where \( n(Si) \), \( n(Cu) \) and \( n(Mn) \) are the concentrations in weight percent of silicon, copper and manganese, and \( a \), \( b \), \( c \), \( d \) and \( e \) are constants determined by theory and/or experiment.

In the plot a carbon concentration of 0.03% by weight is used. With this carbon concentration a general formula of:

\[ T_{Ph} = 1600 - 100n(Si) + 100 (n(Cu) - 0.2) + (200n(Si) - 480) \times (n(Mn) - 0.1) \]

can be given.

It can be easily shown that with minor rearrangements this formula is in the shape of the formula given before.

Several observations can be made from this formula. The phase transition temperature is decreased by an increasing silicon concentration and increased by an increasing copper concentration. The dependence on the concentration of manganese is slightly more complex. For silicon concentrations of less than 2.4% by weight an increasing manganese concentration lowers the phase transition temperature. For a typical grain-oriented magnetic silicon steel the silicon concentration is approximately 3% by weight. In this case the transition temperature is increased with an increasing manganese content.

The formula shows, that within certain limits the phase transition temperature can be adjusted by e.g. increasing the silicon content to compensate for the increase of the phase transition temperature by the addition of copper or manganese.
Although the present invention has been described in detail with reference to the preferred embodiment, the present invention is not limited by the disclosed examples from which the skilled person is able to derive other variations without departing from the scope of the invention.
Claims

1. Method for producing grain-oriented magnetic silicon steel sheets having a high austenite content, preferably larger than 40 %, during hot rolling, including the steps of preparing a melt of steel, hot rolling, high temperature annealing, cold rolling, decarburization and final annealing, wherein the melt of steel contains at least 0.1% by weight of manganese and/or at least 0.2 % by weight of copper and wherein aluminium nitride (AlN) is used as the main inhibitor.

2. Method according to claim 1, wherein the melt of steel contains at least 0.15% by weight of manganese.

3. Method according to claim 1 or 2, wherein the melt of steel contains at least 0.3 % by weight of copper.

4. Method according to one of the preceding claims, wherein the melt of steel contains a low carbon content of at most 0.07% by weight .

5. Method according to one of the preceding claims, wherein the melt of steel contains a low carbon content of at most 0.04% by weight .

6. Method according to one of the preceding claims, wherein the melt of steel contains at most 0.01 % by weight of sulphur.

7. Method according to one of the preceding claims, wherein the melt of steel contains 2.5 % to 6.5 % by weight of silicon.

8. Method according to one of the preceding claims, wherein the melt of steel contains 2.9 % to 3.4 % by weight of silicon.
9. Method according to one of the preceding claims, wherein the melt of steel contains up to 1.2 % by weight of chromium.

10. Method according to one of the preceding claims, wherein at least one cold rolling step with a reduction of over 85% is performed.

11. Method according to one of the preceding claims, wherein at least one of the weight percentages of silicon, manganese and copper in the melt of steel is varied, while keeping the carbon content constant, to achieve a given temperature of the transition $T_{ph}$ from the mixed austenite and ferrite phase region to the pure ferrite phase.

12. Method according to claim 11, wherein the variation is performed by using a formula for the phase transition temperature of the shape:

$$T_{ph} = a - b \cdot n(Si) + c \cdot n(Cu) - d \cdot n(Mn) + e \cdot n(Si) \cdot n(Mn),$$

where $n(Si)$, $n(Cu)$ and $n(Mn)$ are the concentrations in weight percent of silicon, copper and manganese, and $a$, $b$, $c$, $d$ and $e$ are constants determined by theory and/or experiment.

13. Grain-oriented magnetic silicon steel produced by a method according to one of the preceding claims.
### C. DOCUMENTS CONSIDERED TO BE RELEVANT

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