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(11) **EP 1 052 043 A2**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
15.11.2000 Bulletin 2000/46

(51) Int. Cl.⁷: **B22F 3/18, B22F 3/24,
B22F 7/08, C22C 1/04,
C22C 38/02**

(21) Application number: **00109188.3**

(22) Date of filing: **09.05.2000**

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE**
Designated Extension States:
AL LT LV MK RO SI

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(30) Priority: **10.05.1999 JP 12925099
28.12.1999 JP 37235199
14.02.2000 JP 2000035402
14.02.2000 JP 2000035435**

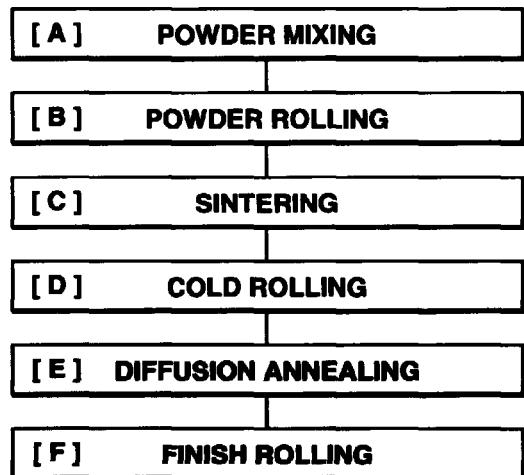
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(54) **Silicon steel plate and method for producing the same**

(57) A silicon steel plate excellent in magnetic properties, workability, flatness, punching quality and the like is produced easily even in a case of high Si content as much as 12 wt%, through [A] powder mixing process using iron powder and Fe-Si alloy powder, [B] powder rolling process for forming the mixture into sheet metal, [C] sintering process, [D] cold rolling process, [E] diffusion annealing process, and [F] finish rolling process for subjecting the sintered, cold-rolled and annealed sheet metal to skin pass rolling.

FIG.1



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Description

BACKGROUND OF THE INVENTION

5 1. Field of the Invention

[0001] This invention relates to a silicon steel plate, which is used for magnetic materials in a shape of plate, strip, hoop, sheet, foil or the like, and excellent in insulation property, corrosion resistance, heat resistance, adhesion, punching quality, magnetostriction property, space factor, alternative magnetic property or so, and further relates to a method
10 of production for such the silicon steel plate.

2. Description of the Prior Art

[0002] As a method for further improving the magnetic steel plate in the insulation property, corrosion resistance,
15 heat resistance, adhesion, punching quality, magnetostriction property, space factor or so, there has been a steel plate provided with an insulating film on the surface thereof.

[0003] For example, in a case of applying the magnetic steel plate (strip) to an iron core of the electric motor or so, the magnetic steel plate is punched out into core disks with predetermined shapes after strain relieving annealing. The laminated iron core is used, which is made by stacking up predetermined number of the core disks and fixed them
20 together in the stacked state through welding, caulking, adhesion or so.

[0004] In this case, an electrical insulating film is formed in the surface of the magnetic steel plate (strip), and the film of this kind is required to be excellent in the corrosion resistance, adhesion, solvent resistance, heat resistance, seizure resistance, oil resistance, slidability after annealing, punching quality, weldability, space factor, auto-caulkability and so on in addition to the insulation property.

[0005] Conventionally, as the insulating film of the magnetic steel plate (strip) of this kind, films of inorganic type, organic type, inorganic-organic mixed type and so have been used, generally the inorganic films have a tendency to be excellent in the slidability after annealing, but not excellent in the punching quality as compared with the organic and the mixed types, and the organic films are excellent in the punching quality and the adhesion.

[0006] There has been many prior arts for forming such the insulating films on the surface of magnetic steel plates
30 (for example, Japanese Patent Application First Publication (Kokai) No.5-44051/93, No.5-26326/93, No.6-65753/94, No.6-145999/94, NO.6-184763/94, No.6-184764/94, No.7-41913/95, NO.7-62551/95, No.7-207424/95, No.8-41650/96, No.9-157861/97, No.10-1779/98, NO.11-12756/99, No.11-71683/99 and so on), however there is a problem in that it is difficult to closely control the adhesion, the punching quality, the space factor or the like of the insulating films.

[0007] The other side, high silicon steel plates containing Si of 2.0 to 4.0 wt% are used as soft magnetic materials
35 for iron cores of transformers or electric motors.

[0008] The most important thing among the properties required in such the silicon steel plates is to be low in core loss so as to decrease energy loss, to improve efficiency and prevent thermal elevation in the electric apparatuses and so on, and the requirement for high-Si materials with higher electric resistance becomes higher in order to improve eddy-current loss for the high-frequency apparatuses in recent years.

[0009] There has been proposed various techniques until now for decreasing core loss by introduction of the high-silicon steel plate and improving the alternative magnetic properties.

[0010] The core loss in the high-silicon steel plate can be considered by dividing into direct current core loss and eddy-current loss, and the eddy-current loss is energy loss according to Joule heat caused by induction.

[0011] The eddy current becomes larger in proportion to changing speed of magnetic flux density with passage of
45 time, therefore, becomes larger with increase of frequency of the alternative current.

[0012] The electric resistance of steel becomes larger by adding Si into the steel plate, thereby enabling decrease of the eddy current. Accordingly, Si has been contained in magnetic steels until now.

[0013] The reduction of eddy current has been tried by decreasing thickness of the steel plate, by forming a film with different thermal expansion coefficient on a surface of the steel plate in order to give tension on the surface of the steel plate, by refining crystal grains in order to decrease width of magnetic domain and so on in addition to increase of Si
50 content so as to increase the electric resistance.

[0014] Grain oriented magnetic steels are steels of which $\langle 100 \rangle$ orientation of the crystal grain, that is the axis of easy magnetization is directed to a magnetizing direction. Among them, the steel of which grains directed to $(110)001$ orientation (the so-called "Goss orientation") are arranged uniformly in the rolling direction of the steel plate is called as
55 a unidirectional grain oriented magnetic steel plate, and manufactured by using secondary recrystallization.

[0015] Furthermore, the unidirectional grain oriented magnetic steel plate of which Goss orientation is developed is magnetized mainly by movement of 180° -magnetic wall, thereby improving the soft magnetic properties of the steel plate.

[0016] Meanwhile, it is considered to reduce the width of 180° -magnetic domain in order to decrease the eddy-current loss in the high-silicon steel plates because it has been known that the eddy-current loss becomes larger when moved distance of the 180° -magnetic wall becomes larger by increasing the crystal grain size, and the techniques has been investigated for subdividing the 180° -magnetic domain.

5 **[0017]** For example, it is devised to form grooves periodically in a direction perpendicular or inclined within a range of 20° against the rolling direction of the steel plate in Japanese Patent Application First Publication (Kokai) No.5-222490/93. It is disclosed to form the grooves by applying laser beams or by etching with acids.

[0018] Further, it is disclosed to provide sticking layers consisting of oxides, chlorides and sulphides of Sn and/or B on linear regions arranged plurally in the direction substantially perpendicular to the rolling direction of the steel plate
10 after cold rolling in Japanese Patent Application First Publication No.6-65644/94.

[0019] Further, a production method of silicon steel plate is disclosed in Japanese Patent Application First Publication No.6-100997/94, which consists of forming grooves with maximum depth of 2~50 μm in average with spaces in the surface of the steel plate after the primary recrystallization annealing and subjecting the steel to final annealing after coating annealing separation agent.

15 **[0020]** Furthermore, a method is disclosed in Japanese Patent Application First Publication No.6-100393/94 for forming linear grooves extending in the perpendicular direction to the rolling direction in the finally rolled steel plate before the finish annealing, and then filling up Sn, B, Sb, oxides or sulfates of these elements in the linear grooves.

[0021] Additionally, it is described in Japanese Patent Application First Publication No.7-331333/95 to form a large number of groove extending in the direction crossing with the rolling direction in the surface of the steel plate, and form
20 low-Si regions of which Si content is lower than that of material steel by 0.3 wt% or above in a depth of 50 μm or more in respect to at least one of bottom and both side faces of the linear grooves.

[0022] In the aforementioned production method of the silicon steel plates which are low in the eddy-current loss and excellent in the magnetic properties, it is necessary to form grooves in the surface of the high-silicon steel plates which are molten in the furnace and subjected to hot and cold rolling, and there is a problem in that many steps are
25 required for obtaining such the silicon steel plates.

[0023] Furthermore, although such the object can be achieved in the grain oriented silicon steel plate by forming the lines or grooves in the direction perpendicular to the rolling direction of the steel plate, it is not possible to reduce the core loss even by forming the line or grooves on the surface of the steel plate in the non-oriented silicon steel plate.

[0024] In addition to the above, the high-silicon steel plates containing Si of 6.5 % (magnetostriction = 0) are well
30 known as magnetic materials further suitable for the iron core of the transformer.

[0025] Furthermore, there is a requirement of magnetic materials excellent in high-frequency properties in the high magnetic flux density in the view point of a recent tendency of miniaturization, improvement of efficiency and increase of frequency in the electric and electronic apparatuses.

[0026] As a material having properties possible to satisfy the aforementioned requirement, a silicon steel plate is
35 well known, which contains Si of 11.5 % as the upper limit value. However, in Fe-Si series alloys, workability of alloys becomes lower according to increase of Si content and cold rolling becomes very difficult if the Si content exceeds 4.5 %.

[0027] When the steel plates of this kind are manufactured through the conventional melting-rolling process, alloying compositions cannot but be selected within the limits of the possibility of rolling, accordingly the so-called "siliconizing" has been proposed as an endeavor for exceeding such the limits at any rate. In this method, a thin plate is made
40 by rolling an alloy excellent in workability, such as Fe-3%-Si alloy, for example, and then Si content in the surface of the plate is increased through CVD method by using SiCl₄, subsequently the amount of Si is unified into approximately 6.5 % by diffusing Si at the surface through successive heating. In this technique, the toxic gas in used and measures against the gas leakage is required in equipment and facilities. Therefore, increase in the cost is not avoidable in both
45 the equipment cost and operation cost in order to sufficiently take measures against an accident.

[0028] As another method, attainment of the steel plate containing a large amount of Si is tried by means of powder metallurgy. However, the obtained plate is low in the workability owing to the high Si content and difficult to be subjected to the cold rolling even in this method, and there is also a limit in the view point that it is impossible to obtain the steel plate with a desired thickness.

50 **[0029]** As the other competitive processes, there is a technique of mixing fine powder of Fe-Si alloy with a appropriate binder and rolling the mixture after making its thickness uniform with a doctor blade. The process is expensive in the cost because especially fine powder is required and the binder is used. The binder has to be removed during the process, and it takes a long time to remove the binder. Furthermore, there is a restriction to execute sintering at elevated temperature, so that it is unsuitable process for mass production of the thin plate of silicon steels.

55 **[0030]** There is also a measure of containing powder into a can made of material excellent in workability such as mild steels and hot-working the can contained with the powder after sealing. However, it is necessary to remove the can after the working, thereby causing increase of the cost. Further, cold rolling is not available after the hot working, therefore it is not possible to obtain a thin plate with a thickness of 0.5 mm and below.

SUMMARY OF THE INVENTION

[0031] This invention is made in order to solve the aforementioned problems of the non-oriented magnetic steel plate in the prior art, for the purpose of providing a silicon steel plate excellent in punching quality and adhesion of the insulating films, a high-silicon steel plate excellent in the magnetic properties, and providing a method possible to easily produce such the silicon steel plate with high Si content.

[0032] That is, the silicon steel plate according to this invention is characterized by being made from metal powder through powder rolling and coated with an insulating film on a surface thereof, preferably after sintering.

[0033] The silicon steel plate according to another embodiment of this invention is characterized by being made from metal powder through powder rolling and diffusion annealing, and roughness caused by metal powder in a surface of the steel plate is controlled through cold rolling and so.

[0034] The production method of the silicon steel plate according to this invention is characterized by coating the insulating film on a surface of a sheet metal obtained by powder-rolling the metal powder containing Si, preferably after subjecting the sheet metal to diffusion sintering prior to the coating of the insulating film.

[0035] The production method of the silicon steel plate according to the other embodiment of this invention is characterized by comprising the steps of powder-rolling the metal powder containing Si into a sheet metal; subjecting the obtained sheet metal to diffusion annealing, and controlling roughness caused by the metal powder in a surface of the sheet metal through cold rolling and so.

BRIEF DESCRIPTION OF THE DRAWINGS

[0036]

FIG.1 is a flow diagram illustrating the basic processes in the production method of the silicon steel plate according to this invention;

FIG.2 is a flow diagram illustrating the simplified processes in the production method of the silicon steel plate according to this invention;

FIG.3 is a flow diagram illustrating the modified processes of the production method shown in FIG.1;

FIG.4 is a flow diagram illustrating the modified processes of the production method shown in FIG.2;

FIG.5 is a flow diagram illustrating the other modified processes of the production method shown in FIG.1;

FIG.6 is a flow diagram illustrating the other modified processes of the production method shown in FIG.2;

FIG.7 is a graph illustrating relationship between sinter parameter P_1 and areal percentage A_1 of a part of which Si content is 5.5 % or below in the sintered body; and

FIG.8 is a graph illustrating relationship between sinter parameter P_2 and areal percentage A_2 of a part of which Si content is 6 % to 7 % in the sintered body.

DETAILED DESCRIPTION OF THE INVENTION

[0037] The silicon steel plate according to this invention is coated with an insulating film on the surface of a sheet metal obtained by powder-rolling metal powder containing Si, or the surface of a sheet metal further subjected to diffusion sintering after the powder rolling of the metal powder.

[0038] In this case, magnetic steel powder, such as Fe-3wt% Si, Fe-4.5wt% Si, Fe-6.5wt% Si, Fe-Si series alloying composition and the like, may be used as the metal powder. Also as the metal powder, pre-mixed powder obtained by mixing powders in advance so as to accord with the chemical compositions of the magnetic steel, pre-alloy powder partially alloyed in advance and having the chemical compositions of the magnetic steel and the like may be used. It is possible to sufficiently utilize characteristics of iron powder excellent in moldability by using a powder mixture of the iron powder and Fe-Si alloy powder containing Si of 8 to 65 wt%.

[0039] In the insulating film to be coated on the surface of the sheet metal obtained by powder-rolling the aforementioned metal powder (steel, iron and/or alloy powders), inorganic matter is used, such as slurry-like material containing MgO, SiO₂, Al₂O₃, ZrO₂, SnO₂, TiO₂, CrO₃, B₂O₃, Mg₂SiO₄ or the like, phosphoric acid and phosphates, chromic acid and chromates, boracic acid and borates or so.

[0040] Organic matter is also used, such as acrylic resins, alkyd resins, phenol resins, epoxy resins, melamine resins, silicone resins, amino resins, styrene resins, ethylene resins, polyvinyl chloride resins, polyvinyle acetate resins, isocyanate resins, polyester, polyamido, polystyrene, polypropylene, polycarbonate, polyurethane, or so.

[0041] Furthermore, a mixture of the inorganic and organic matter or two-layer structure composed of the inorganic and organic matter may be also used.

[0042] Although roughness is formed on the surface of the sheet metal owing to use of the metal powder, a pitch of the roughness may be controlled by selecting particle size of the metal powder. Further, the depth of the roughness may

be controlled by selecting the reduction ratio at the time of powder rolling or selecting the sintering temperature at the time of diffusion sintering (diffusion annealing) of the sheet metal.

[0043] Furthermore, the sheet metal obtained through the powder rolling may be subjected to cold rolling, warm rolling at a temperature lower than recrystallization temperature, or hot rolling after the sintering, and the depth of the roughness caused on the surface of the sheet metal owing to use of the metal powder may be controlled by selecting the reduction ratio at the time of the rolling.

[0044] In the silicon steel plate according to this invention, the insulating film is formed on the surface of the sheet metal after forming by the powder rolling, after subjecting the sheet metal to the diffusion sintering or after further subjecting the sintered sheet metal to rolling such as the cold rolling. The insulating film can be formed in a single layer or double layers by selecting among the aforementioned various substances.

[0045] In this manner, the insulating film is not limited only in the single layer and it is possible to form, for example, Mg-Si complex oxide, such as forsterite (Mg_2SiO_4) on the powder-rolled Fe-3% Si magnetic steel sheet as a lower layer, and further form Cr-oxide and epoxy resin on the phorstellite layer as an upper layer.

[0046] There are various methods for forming the insulating film on the powder-rolled sheet metal, and the forming method of the insulating film is not limited in the specific method in this invention. For example, a brushing method, a spraying method, a dipping method and the like are available.

[0047] The thickness of the insulating film is suitable to be 0.5 to 5 μm or so, and the applying amount of the insulating film is suitable to be 0.5 to 3.0 g/m^2 or so, however it is desirable to select the thickness and the applying amount in accordance with the coating method, the type of the film, the number of the coating layer and so on.

[0048] In the silicon steel plate provided with the insulating film and obtained in this manner, the roughness is formed on the surface of the powder-rolled sheet metal, powder-rolled and sintered sheet metal or further cold-rolled sheet metal because the metal powder is used, accordingly the adhesion of the insulating film on the surface of the silicon steel plate (sheet metal) becomes excellent remarkably.

[0049] Further in such the powder-rolled silicon steel plate, the insulating property becomes satisfactory by forming the insulating film, the slidability after annealing and the punching quality are also improved. The corrosion resistance, oil resistance, solvent resistance, rusting resistance and so are further improved, and the magnetostriction is reduced by the tension caused by the insulating film.

[0050] In a case where the magnetic steel powder of Fe-6.5wt% Si steel is used as the metal powder, it is not necessary to cope with the magnetostriction by cancelling the tension with the insulating film since the magnetostriction of the magnetic steel containing Si of 6.5 wt% is zero, so that the high-tension film becomes unnecessary to be formed.

[0051] Furthermore, there is the roughness caused by the metal powder on the surface of the powder-rolled or further sintered sheet metal as mentioned above, therefore even when gas is generated by vaporization of the organic substances composing the insulating film at the time of welding, the gas escapes through the roughness parts on the surface of the sheet metal (lower face of the insulating film), so that defects such as blistering, peeling or so are never caused and the weldability is improved very remarkably. Consequently, the space factor becomes higher at the time of pile up the electric steel plates.

[0052] In the silicon steel plate according to another embodiment of this invention, which is made from the metal powder through powder rolling and diffusion annealing, and of which roughness caused by the metal powder in the surface thereof is controlled through cold rolling and so, roops of magnetic flux is formed between pitches of the roughness parts caused by the metal powder as the material, so that magnetic domain is fractionated sufficiently and suitably, the eddy-current loss becomes lower and the silicon steel plate improved in the alternative magnetic properties is provided.

[0053] As chemical compositions of the silicon steel plate, the Si content is preferable to be 5 to 12 wt%. Namely, in the case where the Si content of the silicon steel is less than 5 wt%, the steel is possible to be produced through the conventional rolling process using the ordinary ingot steel and there is not so many merits obtained by introducing the powder rolling process for the production of the steel, so that it is not desirable to apply the powder rolling in the production of the steel containing Si less than 5 wt%. Meanwhile, the Si content in the steel is not desirable to exceed 12 wt% because electric resistance of the steel becomes lower in addition of decrease of the saturated magnetism and the high-frequency property is remarkably degraded.

[0054] As the metal powder used in the powder rolling, powder of Fe-5 to 12 wt% Si alloy may be use, besides mixed powder may be also used, which is a mixture of iron powder excellent in the moldability and Fe-Si alloy powder containing Si of 8 to 65 wt% and mixed in a ratio so as to obtain the powder-rolled silicon steel plate with Si content of 5 to 12 wt% after the diffusion annealing.

[0055] Such the iron powder with the desirable Si content and the high-silicon steel powder are manufactured by the reduction method, the pulverization method, the water atomizing method, the mist atomizing method or the like, and used after adjusting the particular size appropriately. The pre-mixed powder obtained by mixing in advance, the pre-alloy powder alloyed in advance or the like may be used, and it is suitable to use the fine and irregular shaped metal powder with particle size of under 100 mesh.

[0056] The iron powder and the high-silicon steel powder are subjected to the powder rolling and further subjected

to the diffusion annealing in a non-oxidative atmosphere, thereby obtaining annealed plate (solid) from the high-silicon steel powder.

[0057] It is desirable to carry out the diffusion annealing at a temperature of 1150 °C or above.

5 **[0058]** After the diffusion annealing, the roughness formed on the surface of the powder-rolled and annealed sheet metal during the sintering process of the metal powder by subjecting to cold rolling, thereby controlling the fractionation of magnet domain.

[0059] In this case, the pitch of the roughness on the surface may be controlled by regulating the particle size of the metal powder.

10 **[0060]** The depth of the roughness on the surface of the sheet metal also may be controlled by regulating annealing temperature at the time of the diffusion annealing.

[0061] Similarly, the depth of the roughness can be controlled by regulation of reduction ratio at the time of the cold rolling.

[0062] The rolling has also the advantage in that it is possible to improve the space factor of the silicon steel plates piled up so as to be used for the iron core or so as compared with the case in which the rolling is not performed.

15 **[0063]** In the method for producing the silicon steel plate according to the other embodiment of this invention, although the powder rolling of the metal powder, the diffusion annealing of the powder-rolled sheet metal, the rolling and so are carried out, the method may be divided into a basic form and a simplified form.

20 **[0064]** The basic method for producing the silicon steel plate according to this invention comprises, as shown in FIG.1, the processes of [A] powder mixing process, [B] powder rolling process, [C] sintering process, [D] cold rolling process, [E] diffusion annealing process and [F] finish rolling process.

[0065] Furthermore, the simplified method for producing the silicon steel plate according to this invention comprises, as shown in FIG.2, the processes of [A] powder mixing process, [B] powder rolling process, [E'] diffusion annealing process and [F] finish rolling process.

25 **[0066]** Various modification and variation can be applied to the respective cases of the aforementioned basic and simplified forms of the production method of the silicon steel plate.

[0067] As the first example, the following process [G] may be performed successively after the finish rolling process [F];

30 [G] flattening treatment process for annealing the sheet metal in a state of applying tension in the longitudinal direction (the so-called "tension annealing").

[0068] The flow diagram in the case of adding the flattening treatment process [G] to the basic method shown in FIG.1 is shown in FIG.3, and the flow diagram in the case of adding the flattening treatment process [G] to the simplified method shown in FIG.2 is shown in FIG.4.

35 **[0069]** As another example of the modification, any one of the following processes [H] and [I] may be carried out at least one time in advance of the diffusion annealing process [E] or [E'], especially in a case where the thin plate is intended to be obtained with high dimensional accuracy;

40 [H] combination of cold rolling and successive process annealing for heating the sheet metal at a temperature of not lower than 600 °C and lower than 950 °C; and

[I] warm rolling at a temperature of not lower than 600 °C and lower than 900 °C.

[0070] The flow diagram of the modified method in which cold-rolling and annealing [H] is executed in addition to the basic method shown in FIG.1 and the aforementioned flattening treatment process [G] is shown in FIG.5.

45 **[0071]** As the variation of the simplified method shown in FIG.2, it is recommended to execute the following process [H] once in advance of the diffusion annealing process [E];

[H] combination of annealing for heating the sheet metal at a temperature of not lower than 600 °C and lower than 950 °C, and successive cold rolling.

50 **[0072]** The flow diagram of the modified method in which annealing and cold rolling [H] is performed in addition to the simplified method shown in FIG.2 is shown in FIG.6.

55 **[0073]** As iron powder used for powder material, it is suitable to use the so-called reduced iron powder and atomized iron powder. The iron powder manufactured from iron carbonyl compounds is not suitable because it has excessively fine grain size and nearly spherical shape, and poor in the moldability in addition to its high price. As Fe-Si alloy powder, it is suitable to use powder manufactured by spraying water against the molten alloy. As to the particle size of these iron and Fe-Si alloy powders, it is suitable to use the powder comprising fine and irregular shaped particles possible to pass 100 mesh or so. Two kinds of material powders to be mixed are desirable in the average and distribution

of the particle size. If they are different remarkably from each other, there is the possibility that the two kinds of powders separates from each other during the handling of the mixed powder.

[0074] The diffusion annealing of the powder-rolled metal is carried out in the non-oxidative atmosphere, such as an atmosphere of argon, nitrogen, hydrogen or so, or in vacuum.

5 **[0075]** The aforementioned respective processes in the basic production method have the following significance as explained below.

[0076] That is, in the powder mixing process, low moldability of the Fe-Si alloy powder with high Si content is improved by mixing the iron powder excellent in the moldability so as to carry out the powder rolling under the high moldability in the whole body of the mixture.

10 **[0077]** The sintering of the sheet metal formed by the powder rolling enables the product to be obtained as a result of the succeeding cold rolling to exhibits strength at the same time of maintaining the workability as the mixture. The cold rolling is carried out in order to realize desired thickness and increase the bulk density of the sintered body by smashing holes in the sintered body and giving internal strain energy, whereby it is possible to mitigate the condition in the diffusion annealing of the next process. In this time, "cold rolling" means the rolling in the temperature range at
15 which recrystallization is never caused.

[0078] The cold-rolled sheet having an increased density in this manner is uniformed in the composition and promoted to be compacted by the diffusion annealing, thereby exhibiting intended magnetic properties. The obtained sheet metal is rolled into the predetermined thickness by the finish rolling.

20 **[0079]** The skin pass rolling for finish has merits of not only improvement of accuracy in the thickness of the silicon steel plate to be obtained, but also improvement of flexibility of the product. The improvement of the flexibility is an unexpected profit, the advancement of the workability improves the punching quality and enables to manufacture the product with complicated or minute shape.

[0080] The simplified method is a production method for proceeding microstructural uniformization of the alloying compositions by diffusion, revelation of strength of the sheet metal material and improvement of the magnetic properties at the same time according to the diffusion annealing by heating at a large sinter parameter than that of the sintering
25 process.

[0081] The all cases of the aforementioned production methods, concrete conditions in the respective processes should be selected so as to conform to the aforementioned intention. Although Si content of the Fe-Si alloy powder to be mixed with the iron powder can be selected from the wide range of 8 to 65 wt%, excessive or too little content of Si
30 is not suitable. For example, alloy powder containing Si of less than 8 % is not suitable for manufacturing 6.5 % Si steel, especially in a case of manufacturing thin plate of the steel because the oxygen content which is harmful to the magnetic properties is apt to become higher and the amount of iron powder required for adjusting the compositions of the steel is decreased extremely, thereby degrading the moldability of the powder mixture. Contrarily, alloy powder containing too much Si is also not suitable since the blending ratio of the Fe-Si alloy powder against the iron powder becomes
35 lower and it is difficult to obtain uniformity of the powder mixture.

[0082] This invention has a meaning in producing the silicon steel plate containing high Si content, that is Si content higher than 5 % which is difficult to realize through the conventional technique, and Fe-Si alloy is required to contain Si more than certain limitation value, however too much content of Si is also disadvantageous in consideration of the present situation that the upper limit of Si to be contained Fe-Si alloy steel as the magnetic materials is 11 to 12 % in
40 practical application. Concerning the blending ratio of the iron powder and the Fe-Si alloy powder, unbalanced combination such as the ratio 95:5 or above by weight is not desirable from a view point of ensuring the uniformity of the mixture, and it is preferable to select the ratio 90:10 and further preferable to select the ratio close to the ratio 50:50.

[0083] In order to the above conditions, Fe-Si alloy powder of which Si content is more than 10 % and does not exceed 30 % so remarkably can be used easily in general. There is a eutectic point (Fe₃Si, melting point : 1200 °C) at
45 a point of 18 % Si in the Fe-Si alloy series, accordingly, it is advisable to use powder of the above-mentioned eutectic alloy.

[0084] As to the combination of the two kinds of powders, various cases of the combination are selectable between the following examples on two extremes:

- 50 (1) large amounts of iron powder + large amounts of Fe-Si alloy powder;
(2) small amounts of Fe-Si alloy powder + large amounts of Fe-Si alloy powder.

[0085] From view points of economical efficiency and moldability of the powder mixture, the former is rather advantageous. Especially in a case of obtaining the thin plate, it is recommended to use the iron powder in the large quantity
55 in order to ensure the moldability. It is facilitated to realize the uniform alloying compositions by using the powder mixture of which deviation of the microscopic compositions is smaller, that is the later combination of the powders. In addition to the above, it is necessary to regard the value of Si content of the silicon steel plate to be manufactured as important, and the concrete combination of the chemical compositions should be decided by considering these various

factors synthetically.

[0086] The sintering process is a process for the purpose of obtaining sintered body of which holes is easy to be smashed in the succeeding cold rolling process without promoting diffusion so much. The sintering does not proceed in the practical speed at a temperature lower than 950 °C of the lower limit of the sintering temperature range, and the upper limit of 1400 °C is set because the alloy powder is molten at a temperature higher than 1400 °C.

[0087] This sintering process should be executed under conditions that areal percentage of the part of which Si content is 5.5 % or below is in a range of 30 to 80 % in order to ensure the workability at the proceeding process as mentioned above. "The part of which Si content is 5.5 % or below" is, of course, a part holding the workability, therefore if the sintering is advanced until the areal percentage of this part becomes lower than 30 %, the workability is remarkably degraded in the successive process. The other side, in the sintering such that undiffused part remains as much as above 80 %, the sintered body is insufficient in the strength and the rolling work becomes difficult in itself. It is possible to measure the Si content by EPMA (Electron Probe Micro-Analyzer) as well known by the person having ordinary skill in the art.

[0088] In this stage, on the way to sintering, a large number of the holes exist in the sheet metal, so that the diffusion rate is high and sinter parameter P_1 is expressed as following equation;

$$P_1 = T \times (20 + \log_{10} t)$$

wherein

T: absolute temperature
t: time (min)

[0089] It has been found that relationship between the sinter parameter P_1 and the areal percentage of Si-diffusion can be rearranged in comparatively good order as shown in FIG.7.

[0090] In FIG.7, value of the sinter parameter P_1 for setting the areal percentage A_1 of "the part of which Si content is 5.5 % or below" in the aforementioned optimum range of 30 to 80 % is in a range of $(230\sim310) \times 10^2$, and sintering conditions corresponding to such the sinter parameter value are 950 °C \times 30 min and 1350 °C \times 10 min, respectively. Therefore, it is possible to select the actual operating condition as combination of the temperature and time within the aforementioned range. Especially desirable value of sinter parameter P_1 is within a range of $(270\sim280) \times 10^2$ approximately as is apparent from FIG.7.

[0091] As against the aforementioned sintering, the diffusion annealing is a process for contriving to uniformize the compositions by diffusion of Si and directing the increase of the density, therefore it is necessary to heat at a high temperature of 1150 °C or more. Although the diffusion proceeds in some degree even in a temperature lower than above, it is not possible to expect the increase of the density and the magnetic properties of the steel plate product is not improved, the effect on the improvement of the magnetic properties becomes higher according as the heating temperature is raised, but is saturated in the region of 1350 °C. The alloy is molten at a temperature higher than 1400 °C. The diffusion annealing can be performed either in the batch furnace or the continuous furnace, however it is necessary to apply the anti-seizure agents such as almina for fear that seizure may be caused in the works overlapping one another in a case of using the batch furnace.

[0092] This diffusion annealing process should be executed under conditions that a part of which Si content is 6 to 7 % amount to 50 % or more in areal percentage, particular grains are not coarsened excessively and workability can be ensured in the following process. Sinter parameter P_2 in the diffusion annealing stage is expressed as following equation;

$$P_2 = T \times (10 + \log_{10} t)$$

wherein

T: absolute temperature
t: time (min)

[0093] It has been found that relationship between the sinter parameter P_2 and the areal percentage of Si-diffusion can be rearranged as shown in FIG.8.

[0094] In the FIG.8, value of the sinter parameter P_2 for setting the areal percentage A_2 of "the part of which Si content is 6 to 7 %" in the optimum range of 50 % or more is in a range of $(170\sim200) \times 10^2$, and sintering conditions corresponding to such the parameter value are 1200 °C \times 30 min (or 1150 °C \times 60 min) and 1350 °C \times 120 min, respectively. Accordingly, the actual operating condition may be selected from the combination of the temperature and time within

the aforementioned range. Desirable value of sinter parameter P_2 is especially in a range of $(180-200) \times 10^2$ approximately as is apparent from FIG.8.

[0095] The annealing process is an operation for facilitating the following cold rolling by relieving the strain caused by rolling, and it is not possible to improve the strength of the product in this process. The relieving of work-strain does not proceed at a temperature lower than 600 °C, however even if the annealing temperature is raised at 950 °C or above, further improvement of the workability can not be expected any longer and the energy is merely dissipated wastefully.

[0096] According to the above-mentioned method, it is possible to obtain the silicon steel plate of which Si content is 5 to 12 wt%, thickness is 0.05 to 0.50 mm (practical thickness is in a range of 0.10 to 0.35 mm), chemical compositions are uniform and workability is excellent, and the product according to this production method is also included in the scope of this invention.

EXAMPLE 1

[0097] Next, the invention will be explained in detail on basis of following examples, needless to say, this invention is not limited only in these examples.

[0098] The silicon steel plates were made by using two kinds of metal powder as raw materials, and the two kinds of powder mixtures of Fe-3.5wt% Si powder and Fe-6.5wt% Si powder were prepared.

[0099] First of all, the metal powder was charged into the hopper from the upper part, and powder-rolled sheet metal was formed by subjecting the metal powder supplied successively from the bottom part of the hopper to the powder rolling, and then the powder-rolled sheet metal was subjected to primary sintering at a temperature of 700 °C, subsequently subjected to secondary sintering at a temperature of 1300 °C. Consequently, four kinds of powder-rolled sheet metal having thickness of 0.1 mm and respective surface roughness as shown in Table 1 were obtained by further performing cold rolling, warm rolling and hot rolling in combination.

Table 1

Sheet metal No.	Chemical composition	Surface roughness Ra (μm)	Production method	Remarks
1	Fe-3.5wt% Si	0.31	Powder rolling + Sintering	Inventive Example
2	Fe-3.5wt% Si	0.63		
3	Fe-6.5wt% Si	0.30		
4	Fe-6.5wt% Si	0.61		
5	Fe-3.5wt% Si	0.29	Melting + rolling (Ingot steel)	Comparative Example
6	Fe-6.5wt% Si	0.32	Rolling + CVD	

[0100] Next, two kinds of insulating films shown in Table 2 were applied on the respective sheet metal shown in Table 1 through the roll coating method and put together by baking under conditions shown in Table 2 after drying.

[0101] In this time, two kinds of sheet metal manufactured by melting method (ingot steel) and rolled into 0.1 mm thickness and having Si content of 3.5 wt% and 6.5 wt% were also used as comparative examples.

Table 2

Insulating film No.	Ingredients		Amount of application (g/m ²)	Baking temperature (°C)
1	Chromic acid	45wt%	2	450
	Boric acid	10wt%		
	Magnesium oxide	15wt%		
	Ethylen glycol	10wt%		
	Epoxy resin	20wt%		

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Table 2 (continued)

Insulating film No.	Ingredients	Amount of application (g/m ²)	Baking temperature (°C)
2	Silicone resin	2	300

[0102] Evaluation of various characteristic were carried out with respect to the silicon steel plates coated with insulating films according to the above-mentioned process, and the results are shown in Table 3. The respective characteristic of the silicon steel plates shown in Table 3 were evaluated in accordance with the following procedure.

(1) Surface insulation resistance

[0103] The surface insulation resistance was measured according to the method specified in JIS C 2550 (1986) "Methods of Test for Electrical Steel Sheets"

(2) Adhesion

[0104] The adhesion was indicated with the minimum diameter of bent portion at the time when the outer insulating film does not separate from the steel plate by peeling test using the adhesive tape even by bending the silicon steel plate with the insulating film at an angle of 180°.

(3) Corrosion resistance

[0105] The corrosion resistance was evaluated with percentage of rusting area after leaving the silicon steel plate with the insulating film in an atmosphere of 40 °C - 80 °C RHD for 100 hrs.

(4) Punching quality

[0106] The punching quality was indicated with the number of punchings at the time when the height of burr comes up to 50 μm at clearance of 5 % using the steel die of SKD-1 (alloy tool steel containing Cr). The test was discontinued at 2 million times which are the practical life time.

Table 3

No.	Sheet metal (Table 1)	Insulating film (Table 2)	Evaluation of characteristics				
			Surface insulation resistance ($\Omega \cdot \text{cm}^2/\text{sheet}$)	Adhesion ($\text{mm}\phi$)	Corrosion resistance (%)	Punching quality ($\times 10^4$ times)	Space factor (%)
Inventive example	1 (3.5 wt% Si)	1	40	10	0.5	>200	99.2
	2	2	42	9	0.4	>200	99.3
	3 (3.5 wt% Si)	1	45	12	0.6	>200	98.4
	4	2	44	11	0.5	>200	98.5
	5 (6.5 wt% Si)	1	60	14	<0.1	59	99.3
	6	2	63	12	<0.1	58	99.2
	7 (6.5 wt% Si)	1	65	16	<0.1	56	98.4
	8	2	64	14	<0.1	55	98.6
Comparative example	5 (3.5 wt% Si, Ingot steel)	1	38	14	1.2	>200	98.3
	2	2	37	13	1.1	>200	99.4
	3 (6.5 wt% Si, Rolling+CVD)	1	58	19	0.8	18	99.2
	4	2	57	18	0.9	12	99.4

[0107] As is obvious from Table 3, it was confirmed that the silicon steel plate excellent in the insulation property, corrosion resistance, adhesion, punching quality and having high space factor can be obtained according to this inven-

tion.

EXAMPLE 2

5 **[0108]** The high-silicon steel having chemical compositions shown in Table 4 were molten, and then the high-silicon steel powder having particle size distribution as shown in Table 5 were obtained by water atomizing method.

Table 4

C	Si	Mn	P	S	Sol.Al	N	Fe
0.008	11.76	0.08	0.020	0.025	0.025	0.009	Bal .

Table 5

-350	+350/-200	+200/-150	+150/-100	+100/-50
51.7	37.0	11.2	0.1	0

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[0109] Next, a powder preparation with composition of Fe-6.5wt% Si was obtained by mixing the above-mentioned high-silicon steel powder and iron powder, and the powder preparation was powder-rolled into sheet metal of 0.11 mm in thickness, successively the powder-rolled sheet metal was subjected to diffusion annealing at at temperature of 1300 °C as shown in Table 6.

25 **[0110]** Subsequently, the diffusion-annealed (sintered) sheet plate was subjected to cold rolling with respective reduction ratio shown in Table 6, and three kinds of high-silicon steel plates with a thickness of 0.10 mm were obtained by further subjecting to tension annealing. The results of measuring the pitch of the roughness caused by using the metal powder and core loss are also shown in Table 6.

Table 6

No.		Diffusion annealing temperature (°C)	Cold rolling reduction ratio (%)	Pitch of roughness (mm)	Depth of roughness (mm)
Inventive example	9	1300	3	$\bar{X} = 0.04$	$\bar{X} = 0.030$
	10	1300	5	$\bar{X} = 0.07$	$\bar{X} = 0.023$
	11	1300	8	$\bar{X} = 0.11$	$\bar{X} = 0.012$

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40 **[0111]** Results shown in Table 7 were obtained by measuring core loss of the respective silicon steel plates after subjecting the above-mentioned steel plates to annealing at 950 °C for 1 hour.

Table 7

No.		Core loss (Bm = 1000)			
		1KHz	3 KHz	5 KHz	10 KHz
Inventive example	9	32.3	122.6	238.5	635.6
	10	30.1	112.8	217.4	552.2
	11	19.4	89.4	183.7	531.7

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55 **[0112]** As shown in Table 7, it was confirmed that it is possible to obtain the high-silicon steel plate low in core loss and having improved alternative magnetic property according to this invention.

EXAMPLE 3

[0113] As raw material powders, iron powder and Fe-18% Si alloy powder were manufactured by water atomizing method, and powders passed through a 100 mesh sieve were collected. The respective powders were confirmed that average particle size was 40 μm , approximately. These powders were mixed in the ratio 60:34 by the tumbler so that Si content in the mixture may be 6.5 wt%. Nine kinds of silicon steel plates were manufactured by treating the powder mixture in the following various processes.

[0114] In the powder rolling, the horizontal rolling mill provided with two rolls of 200 mm diameter and 240 mm in length was used, the powder mixture was supplied to the rolling mill through the vibrator plate and the powder rolling was performed at constant pressure of 70 ton in kiss roll method. In the respective processes, the diffusion annealing is executed by using the batch furnace or the continuous furnace, and the tension annealing was carried out in the condition of 750 $^{\circ}\text{C} \times 2$ min and the tension of 3 kg/mm^2 except for special mention of the condition. The finish rolling (skin pass rolling) was carried out in the reduction ratio of 0.5 to 5 %.

Comparative example No.5 (prior art)

[0115]

Powder mixing — Powder rolling (thickness : 0.10 mm) — Sintering (batch furnace, 1250 $^{\circ}\text{C} \times 1$ hour, thickness of endproduct : 0.10 mm)

Inventive example No.12 (FIG.1)

[0116]

[A] Powder mixing — [B] Powder rolling (thickness : 0.11 mm) — [C] Sintering (batch furnace, 1050 $^{\circ}\text{C} \times 1$ hour) — [D] Cold rolling (thickness : 0.105 mm) — [E] Diffusion annealing (batch furnace, 1200 $^{\circ}\text{C} \times 1$ hour) — [F] Finish rolling (thickness of endproduct : 0.10 mm)

Inventive example No.13 (No.12 + [H])

[0117]

[A] Powder mixing — [B] Powder rolling (thickness : 0.12 mm) — [C] Sintering (batch furnace, 1050 $^{\circ}\text{C} \times 1$ hour) — [D] Cold rolling (thickness : 0.11 mm) — [H] Annealing (850 $^{\circ}\text{C} \times 1$ hour) — Cold rolling (thickness : 0.105 mm) — [E] Diffusion annealing (batch furnace, 1200 $^{\circ}\text{C} \times 1$ hour) — [F] Finish rolling (thickness of endproduct : 0.10 mm)

Inventive example No.14 (No.13 + [G], FIG.5)

[0118]

[A] Powder mixing — [B] Powder rolling (thickness : 0.12 mm) — [C] Sintering (1050 $^{\circ}\text{C} \times 1$ hour) — [H] Cold rolling (thickness : 0.11 mm) — Annealing (850 $^{\circ}\text{C} \times 1$ hour) — Cold rolling (thickness : 0.105 mm) — [E] Diffusion annealing (batch furnace, 1200 $^{\circ}\text{C} \times 1$ hour) — [F] Finish rolling (thickness of endproduct : 0.10 mm) — [G] Flattening treatment

Inventive example No.15 (FIG.3)

[0119]

[A] Powder mixing — [B] Powder rolling (thickness : 0.11 mm) — [C] Sintering (continuous furnace, 1050 $^{\circ}\text{C} \times 6$ min) — [D] Cold rolling (thickness : 0.105 mm) — [E] Diffusion annealing (continuous furnace, 1285 $^{\circ}\text{C} \times 8$ min) — [F] Finish rolling (thickness of endproduct : 0.10 mm) — [G] Flattening treatment

Inventive example No.16 (FIG.2)

[0120]

5 [A] Powder mixing — [B] Powder rolling (thickness : 0.105 mm) — [E'] Diffusion annealing (batch furnace, 1200 °C × 1 hour) — [F] Finish rolling (thickness of endproduct : 0.10 mm)

Inventive example No.17

10 [0121]

[A] Powder mixing — [B] Powder rolling (thickness : 0.120 mm) — [H] Annealing (850 °C × 0.5 hours) — Cold rolling (thickness : 0.110 mm) — [C] Sintering (batch furnace, 1200 °C × 1 hour in vacuum) — Cold rolling (thickness : 0.105 mm) — [E'] Diffusion annealing (batch furnace, 1200 °C × 1 hour) — [F] Finish rolling (thickness of endproduct : 0.10 mm)

Inventive example No.18 (FIG.4)

[0122]

20 [A] Powder mixing — [B] Powder rolling (thickness : 0.105 mm) — [E'] Diffusion annealing (continuous furnace, 1285 °C × 8 min) — [F] Finish rolling (thickness of endproduct : 0.10 mm) — [G] Flattening treatment

Inventive example No.19 (FIG.6)

25 [0123]

[A] Powder mixing — [B] Powder rolling (thickness : 0.105 mm) — [E'] Diffusion annealing (continuous furnace, 1325 °C × 2 min) — [F] Finish rolling (thickness of endproduct : 0.10 mm) — [G] Flattening treatment

Inventive example No.20

[0124]

35 [A] Powder mixing — [B] Powder rolling (thickness : 0.11 mm) — [C] Sintering (continuous furnace, 1050 °C × 6 min) — [D] Cold rolling (thickness : 0.105 mm) — [E] Diffusion annealing (continuous furnace, 1285 °C × 8 min) — [F] Finish rolling (thickness of endproduct : 0.10 mm) — [J] Magnetic annealing (batch furnace, 900 °C × 2 hours in vacuum) — [G] Flattening treatment

40 [0125] The density was measured as to the obtained silicon steel plates of comparative example No.5, inventive example Nos.12, 13, 14, 16 and 17, and the specific resistance was further measured as to the steel plates of comparative example No.5 and inventive example No.12. These results are shown together in Table 7.

Table 7

No.		Density ρ (g/cm ³)	Specific resistance ($\mu\Omega\text{cm}$)
Comparative example	5	7.32	80
Inventive example	12	7.31	80
	13	7.38	-
	14	7.42	-
	16	7.36	-
	17	7.33	-

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[0126] The value of the direct current magnetic properties are shown in Table 8.

Table 8

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No.		B1	B2	B10	B25	Hc(Oe)
Comparative example	5	8200	9350	11800	12700	0.32
Inventive example	12	9800	10900	11200	12800	0.25
	13	8750	9780	11850	13010	0.28
	14	8860	9640	11800	12930	0.27
	15	8750	9580	11700	12900	0.38
	16	8700	9480	11500	12700	0.46
	17	8100	9250	11700	12800	0.30
	18	8710	9500	11600	12600	0.52
	19	8680	9480	11300	12600	0.40
20	9630	10540	11800	12900	0.34	

[0127] The alternative magnetic properties are shown in Table 9.

Table 9

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No.		W10/50	W10/100	W10/400	W10/1K	W10/2K	W10/5K	W10/10K
Comparative example	5	0.81	1.86	8.52	33.8	70.3	261.1	714.3
Inventive example	12	0.74	1.84	8.00	31.6	68.6	253.6	710.2
	13	0.85	1.95	9.10	35.5	73.5	280.5	784.4
	14	0.82	1.91	8.86	34.7	71.9	277.9	778.8
	15	0.80	1.90	9.00	36.0	77.8	288.4	804.2
	16	0.88	2.13	10.04	51.1	92.3	341.9	981.7
	17	0.78	1.88	6.81	34.2	70.8	274.9	771.6
	18	0.90	2.15	10.50	52.3	96.6	364.6	1005.9
	19	0.85	2.00	9.80	49.5	87.3	320.9	960.6
20	0.72	1.70	8.10	32.0	70.0	288.0	804.0	

Bm = 10 KG (unit : w/kg)

45 **[0128]** Effect of the flattening treatment was confirmed by comparing flatness of the sample plates cut out from the steel plates of inventive example No. 13 and 14, namely the flatness was compared by measuring the maximum height at the time of placing the sample plates of 85 mm in width and 1 m in length on the horizontal level block. The results are shown in Table 10.

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Table 10

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No.		Flatness (μm/m)
Inventive example	13	836
	14	235

[0129] Furthermore, the flatness to be obtained by the tension annealing was compared in the various conditions

as to the steel plate of inventive example No.15, and the results are shown in Table 11.

Table 11

No.	Tension annealing condition		Flatness (μm/m)
	Temperature (°C)	Tension (kgf/mm ²)	
Inventive example No.15	Not executed	-	1040
	600	4	965
	700	2	784
	700	3	669
	700	4	230
	750	2	320
	750	3	168
	800	3	246
	1000	2	Fractured

[0130] The advantage produced by the skin pass rolling in the finish rolling process [G], that is the improvement of the flexibility is apparent from the following results (shown in Table 12) of the transverse test executed as to the steel plate of inventive example No.12.

Table 12

No.		Transverse rupture strength (MPa)	Deflection (mm)
Inventive example No.	After diffusion annealing	998	0.80
	After skin pass rolling	1986	1.98
Test piece : 3×5×40 mm, Gauge length : 25 mm			

[0131] According to this invention, it is possible to make up for weakness of low workability in the Fe-Si alloy by the excellent workability of the iron powder, and possible to easily manufacture the high-silicon steel plate containing Si of 5 to 12 wt% which has been difficult to be manufactured conventionally. The thickness of the silicon steel plate can be accurately controlled by subjecting to the skin pass rolling in the finish rolling process. The skin pass rolling improves flexibility and workability of the silicon steel and facilitates the punching of the steel plate, and it is favourable to manufacture the iron core of the transformer as main usage of the silicon steel plate. Furthermore, it is confirmed that the silicon steel plate subjected to the tension annealing exhibits the high flatness.

Claims

1. A silicon steel plate made from metal powder through powder rolling and coated with an insulating film on a surface thereof.
2. A silicon steel plate as set forth in claim 1, wherein said steel plate is sintered prior to coating of said insulating film.
3. A silicon steel plate made from metal powder through powder rolling and diffusion annealing, wherein roughness caused by metal powder in a surface of said steel plate is controlled through cold rolling and so.
4. A silicon steel plate as set forth in claim 3, wherein Si content of said steel plate is in a range of 5 to 12 wt%.
5. A silicon steel plate as set forth in claim 4, wherein thickness of said steel plate is in a range of 0.05 to 0.35 mm.
6. A silicon steel plate as set forth in claim 4, wherein said steel plate is made from mixed powder of iron powder and

Fe-Si alloy powder containing Si of 8 to 65 wt%.

- 5
7. A method for producing the silicon steel plate according to claim 1 or 2, characterized by coating the insulating film on a surface of a sheet metal obtained by powder-rolling the metal powder containing Si.
8. A method for producing the silicon steel plate as set forth in claim 7, wherein said sheet metal obtained by powder-rolling the metal powder is subjected to diffusion sintering prior to the coating of the insulating film.
9. A method for producing the silicon steel plate as set forth in claim 7 or 8, wherein said metal powder is magnetic steel powder.
- 10
10. A method for producing the silicon steel plate as set forth in any one of claims 7 to 9, wherein a pitch of roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of particle size of the metal powder to be used.
- 15
11. A method for producing the silicon steel plate as set forth in any one of claims 7 to 10, wherein depth of roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of a reduction ratio at the time of powder rolling and/or sintering temperature at the time of diffusion sintering.
- 20
12. A method for producing the silicon steel plate as set forth in any one of claims 7 to 11, wherein said sheet metal is further subjected to rolling and depth of roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of a reduction ratio at the time of rolling.
- 25
13. A method for producing the silicon steel plate according to any one of claims 3 to 6, comprising the steps of:
- powder-rolling the metal powder containing Si into sheet metal;
subjecting the obtained sheet metal to diffusion annealing; and
controlling roughness caused by the metal powder in a surface of said sheet metal through cold rolling and so.
- 30
14. A method for producing the silicon steel plate as set forth in claim 13, wherein said metal powder contains Si of 5 to 12 wt%.
- 35
15. A method for producing the silicon steel plate as set forth in claim 14, wherein said metal powder is a mixture of iron powder and Fe-Si alloy powder containing Si of 8 to 65 wt%.
- 40
16. A method for producing the silicon steel plate as set forth in any one of claims 13 to 15, wherein a pitch of the roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of particle size of the metal powder to be used.
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17. A method for producing the silicon steel plate as set forth in any one of claims 13 to 16, wherein depth of the roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of annealing temperature at the time of said diffusion annealing.
18. A method for producing the silicon steel plate as set forth in any one of claims 13 to 17, wherein depth of the roughness caused by the metal powder in the surface of said sheet metal is controlled by regulation of a reduction ratio at the time of the cold rolling.
19. A method for producing the silicon steel plate according to any one of claims 3 to 6, comprising the processes of :
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- [A] powder mixing process for obtaining a mixture containing 5 to 12 wt% of Si by mixing iron powder and Fe-Si alloy powder containing 8 to 65 wt% of Si;
[B] powder rolling process for forming the obtained mixture into sheet metal with a thickness of 0.05 to 0.5 mm;
[C] sintering process for sintering the sheet metal obtained through the powder rolling, at a temperature of not lower than 950 °C and lower than 1400 °C;
- 55
- [D] cold rolling process for rolling the sintered sheet metal into a predetermined thickness so as to increase bulk density of the sheet metal to not lower than 98 %;
[E] diffusion annealing process for heating the cold-rolled sheet metal at a temperature of not lower than 1150 °C and lower than 1400 °C in a non-oxidative atmosphere so as to uniformize alloying compositions; and

[F] finish rolling process for subjecting the sheet metal having uniformized alloying compositions through the diffusion annealing to skin pass rolling in a reduction ratio of 0.5 to 5 % into a predetermined thickness.

- 5 **20.** A method for producing the silicon steel plate as set forth in claim 19, wherein said finish rolling process [F] is followed by

[G] flattening treatment process for annealing the sheet metal in a state of applying tension in a longitudinal direction of the sheet metal.

- 10 **21.** A method for producing the silicon steel plate as set forth in claim 20, wherein a following process is executed in advance of said flattening treatment process [G]:

[J] treatment for improving magnetic properties.

- 15 **22.** A method for producing the silicon steel plate as set forth in claim 19, wherein any one of following processes is executed in advance of said diffusion annealing process [E]:

[H] combination of annealing for heating the sheet metal at a temperature of not lower than 600 °C and lower than 950 °C, and cold rolling; and

20 [I] warm rolling at a temperature of not lower than 600 °C and lower than 900 °C.

- 23.** A method for producing the silicon steel plate according to any one of claims 3 to 6, comprising the processes of :

25 [A] powder mixing process for obtaining a mixture containing 5 to 12 wt% of Si by mixing iron powder and Fe-Si alloy powder containing 8 to 65 wt% of Si;

[B] powder rolling process for forming the obtained mixture into sheet metal with a thickness of 0.05 to 0.5 mm;

[E'] diffusion annealing process for heating the sheet metal obtained through the powder rolling, at a temperature of not lower than 1150 °C and lower than 1400 °C in a non-oxidative atmosphere so as to increase density of the sheet metal and to uniformize alloying compositions; and

30 [F] finish rolling process for subjecting the sheet metal having uniformized alloying compositions through the diffusion annealing to skin pass rolling in a reduction ratio of 0.5 to 5 % into a predetermined thickness.

- 24.** A method for producing the silicon steel plate as set forth in claim 23, wherein said finish rolling process [F] is followed by

35 [G] flattening treatment process for annealing the sheet metal in a state of applying tension in a longitudinal direction of the sheet metal.

- 40 **25.** A method for producing the silicon steel plate as set forth in claim 24, wherein a following process is executed in advance of said flattening treatment process [G]:

[J] treatment for improving magnetic properties.

- 45 **26.** A method for producing the silicon steel plate as set forth in claim 23, wherein any one of following processes is executed in advance of said diffusion annealing process [E]:

[H] combination of annealing for heating the sheet metal at a temperature of not lower than 600 °C and lower than 950 °C, and successive cold rolling; and

50 [I] warm rolling at a temperature of not lower than 600 °C and lower than 900 °C.

55

FIG.1

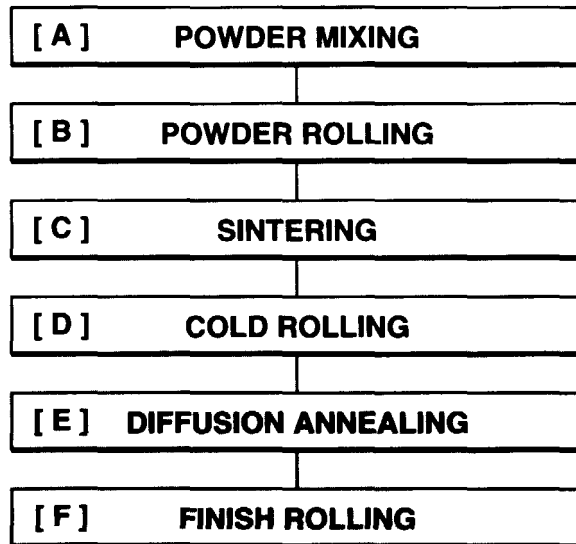


FIG.2

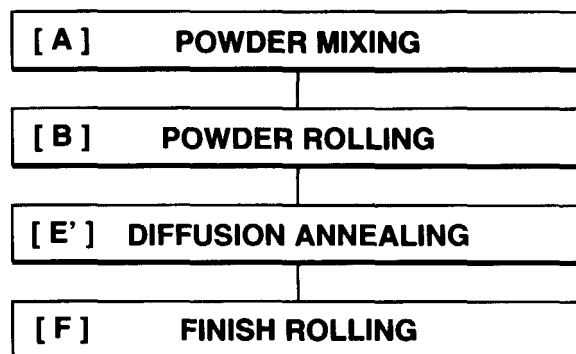


FIG.3

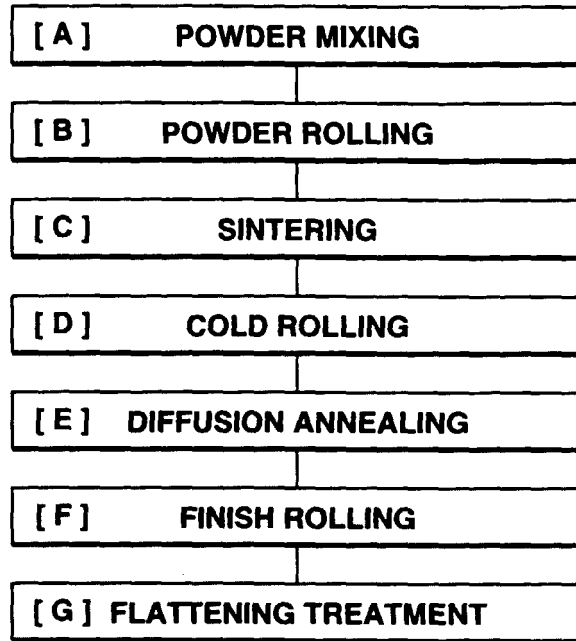


FIG.4

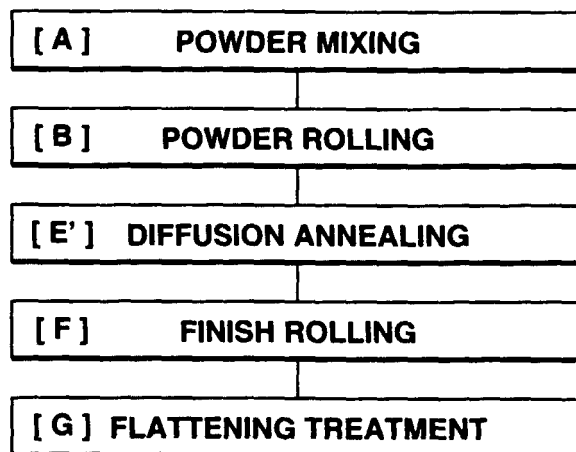


FIG.5

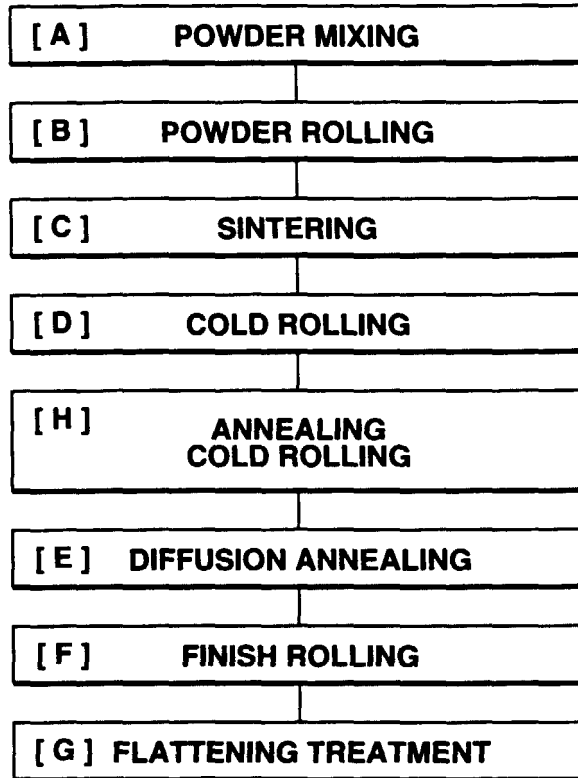


FIG.6

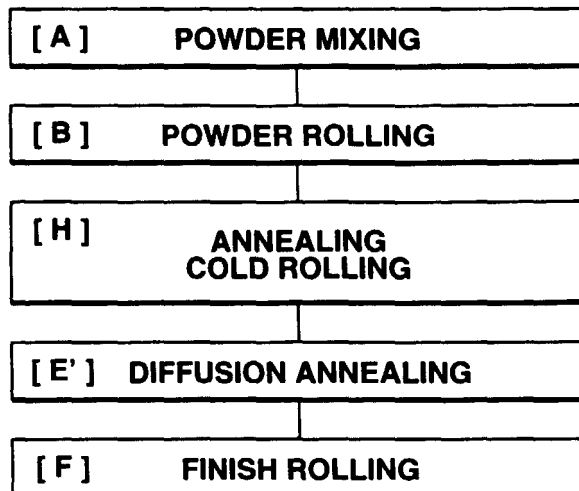


FIG.7

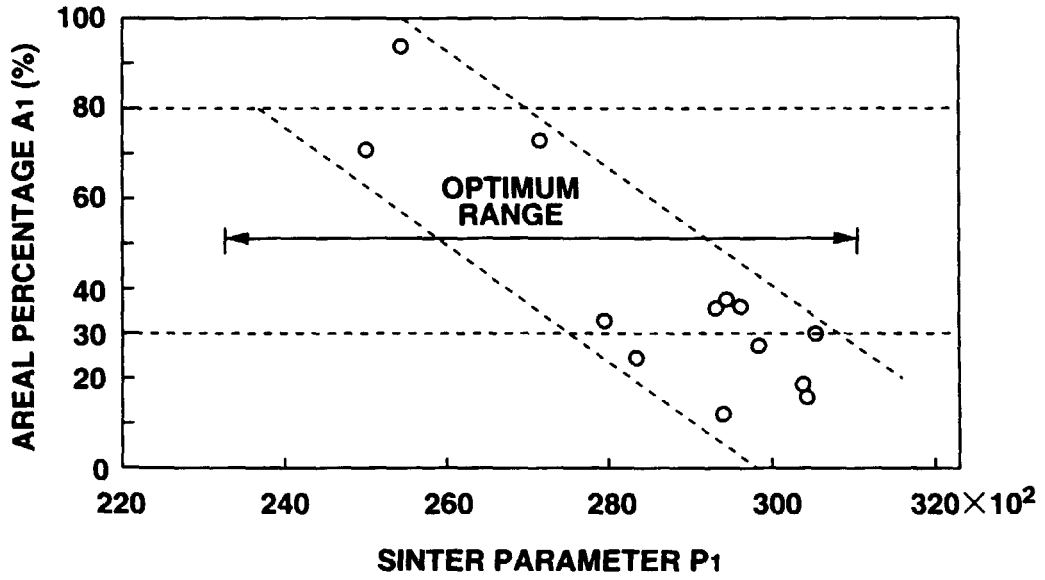


FIG.8

