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(54) **PRODUCTION METHOD OF SOFT MAGNETIC STEEL MATERIAL.**

(57) This invention relates to a method which can produce economically a soft magnetic steel material having high permeability. The production method of the present invention comprises hot working a cast iron billet or steel billet consisting of a pure iron type component as the principal component and 0.5 ~ 2.0 % of Sol. Al and if necessary, 0.005 ~ 1.0 % of Ti at a predetermined heating temperature and at a finish working temperature, and annealing finally at 900 ~ 1,300 °C and preferably at 1,000 ~ 1,300 °C, to obtain a soft magnetic steel material having coercive force of up to 0.4 Oe and a flux density of at least 10,000 G at 0.5 Oe.

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A METHOD OF PRODUCING SOFT MAGNETIC STEEL MATERIALS

TECHNICAL FIELD

The present invention relates to a method of producing soft magnetic ferrous materials, for instance, used as electromagnetic cores or magnetic shielding materials where good DC magnetization properties are required.

TECHNICAL BACKGROUND

Soft irons or pure irons, permalloy or supermalloy have been used as DC electromagnetic iron cores, or magnetic shielding materials or medical appliances, physical machinery, electronic parts or appliances, which have recently been remarkable especially in their demand development. A magnetic flux density at 1 Oe (called as "B₁ value" hereinafter) of the soft iron or the pure iron is about 3000 to 11000 G. It has been used as the magnetic shielding materials or MRI (tomogram diagnosis apparatus by a nuclear magnetic resonance) or those shielding up to a level around several gaussses of magnetic flux, or as electromagnetic iron core materials.

In the usage where the DC magnetization property is important, problems of conventional techniques on the magnetic shielding will be described. The pure iron having a high saturated magnetization has been used to the magnetic shielding of MRI mainly because of its low cost and good performance. Even an O grade (e.g., JIS C 2504 SUYPO) requiring the most severe properties in JIS specification on electromagnetic soft irons specifies a low limit of the B₁ value only to be 8000 G. Thus it is difficult for them to shield a level of the earth magnetism, and a shielding system of a level lower than several gaussses has become bulky. An Fe-Ni alloy known as the permalloy or the supermalloy is sometimes used for more effective shielding. Those materials are possible to shield the magnetism lower than the earth magnetism but they are very expensive, and further their saturated magnetizations are as low as 1/3 to 2/3 of that of the pure iron. For shielding a high magnetic field, their thickness must be increased extremely. A good deal of their use is however difficult from an economical viewpoint.

Taking the above mentioned situations into consideration, some studies have been made on heightening of the magnetic permeability without spoiling the high saturated magnetization of the pure iron materials. They are, for example, methods taught in Japanese Patent Publication No. 63-45443, Japanese Patent Laid Open No. 62-77420 or "Developments of Ultra Thick Electromagnetic Steel Plates" mentioned in No. 5 of vol. 23, (published in 1984) by Japan Metal Society. Each of these methods aims at improvement of the magnetic permeability through accomplishing with coarsening of ferrite crystal grains. However, those technologies limit objects to hot rolled plates of relatively small thickness, or they could not accomplish not lower than 10000 G with the magnetic flux density at 0.5 Oe (called as "B_{0.5} value" hereinafter). Thus they have not been sufficient for the use where a more severe DC magnetization property is appreciated as the present invention.

Up to the present, such materials have not yet been offered that the saturated magnetization is high, and the magnetic permeability is high, that is, the high magnetic flux density is generated in a low magnetic field corresponding to an extent of the earth magnetism. It is an object of the present invention to provide a method which can produce such materials at economical costs.

DISCLOSURE OF THE INVENTION

For solving the above stated problems, the inventors made investigations on industrial pure irons which were typical soft magnetic materials for the DC magnetic field. Through clearing defects thereof, we obtained knowledge under mentioned.

From standpoints for obtaining the high magnetic permeability, following procedures were found to be effective. (1) The addition of Al makes an effective deoxidation possible, improves the magnetic permeability in company with decreasings of an oxygen amount and oxides, and lowers solute N which is harmful to the magnetic permeability by fixing it as AlN precipitates; (2) The addition of a certain necessary amount enables coarsening of finely scattered AlN, reduce bad influences of AlN, and accelerates coarsening of ferrite grains, and each of these effects is profitable to the improvement of the permeability; (3) Especially the addition of more than 0.5% raise transformation temperatures remarkably, or can provide a uniform ferrite phase, and enables annealing at temperatures exceeding 900 °C without introducing strain used by the phase transformation, accordingly. The annealing at high temperatures brings about removal of lattice

strain and the coarsening of the ferrite grains. The improvement of the magnetic permeability of solute Al itself may be also considered, but by synergistic effects thereof, very excellent permeability may be provided; (4) If Ti is added as required, the solute N is preferentially fixed by Ti and attributes to the improvement of the properties, so that an effort is not required for decreasing N content. From a standpoint of holding the saturated magnetization high, followed findings were obtained; (5) The Al addition exceeding 2% should be avoided; (6) If C and N amounts are high, the transformation temperature lowers, or the necessary amount of Al increases. Further, the properties are deteriorated by the increment of the lattice strain by increasings of solute C and N or precipitations of carbides and nitrides. The inventors found upper limits of C and N amounts for avoiding them, and accomplished the present invention.

10 The invention is set forth as follows.

(1) A method of producing soft magnetic ferrous materials characterized by heating not lower than 700°C but not higher than 1300°C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.5 wt%, Mn: not more than 0.50 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol.Al: 0.5 to 2.0 wt%, N: not more than 0.005 wt%, oxygen: not more than 0.005 wt%, and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700°C, and finally annealing at temperatures of 900 to 1300°C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.

(2) A method of producing soft magnetic ferrous materials characterized by heating not lower than 700°C but not higher than 1300°C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.1 wt%, Mn: not more than 0.15 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol.Al: 0.5 to 2.0 wt%, N: not more than 0.005 wt%, oxygen: not more than 0.005 wt%, and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperature of not lower than 700°C, and finally annealing at temperatures of 1000 to 1300°C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.

(3) A method of producing soft magnetic ferrous materials characterized by heating not lower than 700°C but not higher than 1300°C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.5 wt%, Mn: not more than 0.50 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol.Al: 0.5 to 2.0 wt%, N: not more than 0.012 wt%, oxygen: not more than 0.005 wt%, Ti: 0.005 to 1.0 wt% and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700°C, and finally annealing at temperatures of 900 to 1300°C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.

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DETAILED DESCRIPTION OF THE INVENTION

An explanation will be made to reasons for limiting the chemical composition of this invention.

C is preferable to be as low as possible for securing an excellent magnetic permeability as well as N, but an utmost decrease is difficult in industrial production since it causes an extreme cost-up. In view of raising the transformation temperature by Al addition, if the amount of C addition is not controlled to be low, the amount of Al addition should be increased, resulting in lowering the saturated magnetization, which is contrary to the intention of the invention. Therefore an upper limit of C is 0.004 wt%.

Si contributes to the improvement of the magnetic permeability, but the present invention aims at satisfying the magnetic permeability by the Al addition. Rather, an upper limit is 0.5 wt%, preferably 0.1 wt%, paying attentions to a lowering of a saturated magnetization by much addition of Si.

Since Mn deteriorates the DC magnetization property, lower content is desirable, but an extreme lowering causes the cost-up and the increase of N content. Further, this element also suppress a hot brittleness by fixing S. It may be contained 0.5 wt%, preferably 0.15 wt%, as an upper limit within a range that the Mn/S ratio is not lower than 10.

P and S are impurities, and their lower contents are preferable, if not costing up, and their upper limits are 0.015 wt% and 0.1 wt%.

Al is, as said above, the most important element of this invention. That is, Al brings about the fixing of the solute N, the coarsening of AlN, and the raising of the transformation temperature, and as results, thereby expands a ferrite phase region, so that this element enables annealing at high temperatures, thereby to accomplish the coarsening of the ferrite grains and the decerasing of the internal strain. Furthermore, it is assumed that solute Al itself improves the magnetic permeability. Thus, in the present invention, this element must be added for providing the excellent DC magnetization property. Such effects

of Al may be obtained by adding not lower than 0.5 wt% in a value of sol.Al. On the other hand, it is undesirable to add exceedingly 2 wt%, because the saturated magnetization is lowered. Al addition is determined to be 0.5 to 2 wt% in the value of sol.Al.

Similarly to C, N dissolves into the lattice, and creates the lattice strain to deteriorate the DC magnetization properties. It is desirable that N is as low as possible for not producing Al precipitates. This consideration is to make the added Al exist as useful solute Al, and N content should be not more than 0.005 wt%. In the invention, as will be mentioned later, Ti can be added as required, which is a strong nitride former. This is added for decreasing the above said harms of N without controlling a severe upper limit of the N content which may cause a cost-up, and in this case the upper limit of N is 0.012 wt%.

As is apparent from the above mentioned findings, it is desirable to regulate the total amount of N + C for more exactly securing the DC magnetization properties. It is preferable that in a case of no Ti addition, C + N is not more than 0.007 wt%, while in a case of Ti addition, C + N is not more than 0.014 wt%.

Oxygen, similarly to Mn, deteriorates the DC magnetization properties, and especially gives detrimental influences to the magnetic permeability by generating non metallic inclusions. When preparing a molten steel, oxygen must be enough decreased, and an upper limit is specified to be 0.005 wt%.

Ti is the strong nitride former as said above. If adding it 0.005 to 1.0 wt%, it is possible to avoid considerable damages of the DC magnetization property by a fixing solute N even in such materials where N content is not fully decreased, that is, cheap materials. If the N content is relatively low, the generating amount of nitride are low, and the DC magnetization property may be expected to be improved more or less, accordingly. The Ti addition of more than the upper limit deteriorates the DC magnetization property.

A further explanation will be made to conditions for producing steels of the invention.

The present invention employs ordinary hot working conditions for hot rollings, and heats the steel pieces or cast pieces of the above mentioned chemical compositions at the temperatures of not lower than 700 °C but not higher than 1300 °C for the hot working. In the invention, a lower limit of the work ending temperature is determined to be 700 °C, since cost-ups always depend upon increase of deformation resistance at the hot working in accompany with rollings at low temperature range as well as lengthening of time to be taken for the hot working, and rollings at extreme low temperatures possibly cause grain refining by recrystallization during the annealing.

An annealing to be finally performed should be practiced within a range not falling to a transformation temperature, which is decided mainly by the amount of Al addition, and unless practicing at the temperature of at least 900 °C, preferably not lower than 1000 °C, it is not possible to accomplish much excellent DC magnetization property to be intended by the invention. Actually, if adding 0.001 wt% C, 0.0020 wt% N and around 1 wt% Al, the inventive steel is rendered to be a ferrite single phase, and it is therefore possible to carry out the annealing at very high temperatures of not lower than 1100 °C, but since the annealing at temperature ranges exceeding 1300 °C is difficult and it gives rise to the cost-up, the annealing temperatures are determined to be 900 to 1300 °C, preferably 1000 to 1300 °C. The holding times of the annealing are varied in dependence upon the heat capacity of the material, and it is desirable to hold not less than 30 minutes. With respect to the cooling after the heating, in view of introduction of no thermal strain, a slow cooling is desirable. If an attention is paid so that a uniform cooling may be provided, the thermal strain is difficult to be introduced, and in such a case, the slow cooling is not always required.

If the annealing temperature is especially limited with the chemical composition and under the producing conditions specified in the invention, it is possible to produce ferrous materials of high saturated magnetization and $B_{0.5}$ value, that is, excellent soft magnetic properties at the DC magnetic field.

The present invention also includes a case where a hot direct rolling is employed for the hot rolling. The ferrous materials to be produced by the invention includes both of hot worked materials and cold worked materials (including a warm working). The final annealing is therefore irrespective of a case after the hot working or a case after the hot working - cold working. The invention of course includes such case of performing the intermediate annealing on the half way of the hot working or the cold working, or a case of performing each of the above workings in the several steps. The steels at which the invention aims include plates, sheets, bar, wire materials (shape steels), forged materials, etc.

EXAMPLES

Example 1

Table 1 shows chemical compositions of steels used in the invention and comparative examples. Steels A to E were formed into sheets with thicknesses of 1 to 5 mm by hot rolling at 1200 °C from ingots having thickness of 110 mm after melted, wherein steels A to C fall within the inventive chemical composition, and Steels D, E, F and G are comparative. Table 1 shows transformation point when the temperatures were

elevated up to 1300° C at heating rate of 0.5° C/s. The measures of the transformation point tell that the inventive steels have ferrite single phases.

Table 2 shows the DC magnetization properties of the inventive steels and the comparative steels, wherein the annealings were carried out on test pieces obtained from the center parts of the thickness of the hot rolled steels, having an outer diameter of 45 mm, an inner diameter of 33 mm and a thickness of 6 mm for measuring the DC magnetization permeability and the ferrite grain sizes. The annealings herein corresponds to the final annealing defined in the invention. In the annealing, the heating - holding time was set to be 1 to 3 hours, and the cooling rate was set to be a slow cooling of about 100° C/hr.

Table 2 shows Examples in accordance with the invention where No. 1 carried out the annealing at 1100° C on Steel A. In this Example, since the steel has the ferrite single phase due to lowering of the C content and Al addition, the annealing at the high temperature is possible without introducing transformation strain and grains refining by the transformation. Considerable coarsening of not less than 2 mm in the ferrite grain sizes was accomplished by the annealing at the high temperature as 1100° C, and concurrently the lattice strain was removed, so that very excellent properties of $B_{0.5}$ value being around 13000 G and the maximum magnetic permeability exceeding 60000 were obtained.

No. 2 is an Example where the annealing at 1000° C was done on Steel A where the annealing temperature was lower than that of No.1, although the ferrite grain sizes were smaller than those of No.1 as around 0.5 to 1.0 mm, the properties were good as the maximum magnetic permeability being 23900.

Nos.3 and 4 are Examples of Steels B and C. Also herein, the ferrite single phases were made by the Al additions, and in each of them, it was possible to perform the annealings at the high temperatures exceeding 1000° C. By the synergistic effects of coarsening of the ferrite grains and the removal of internal strain, the excellent properties were available as the maximum magnetic permeability being 56000 in No.3, and 37200 in No.4.

In each of the above Examples Nos.1 to 4, the excellent DC magnetization properties of the maximum magnetic permeability being not less than 20000 and the coercive force being not more than 0.4 Oe were accomplished, which not only satisfied enough the properties specified in JIS C 2504 SUYPO but also even $B_{0.5}$ value exceeds 11000 G, and thus enabled to shield the magnetism to an extent produced by the earth.

Nos.5, 6 and 7 are comparative Examples of Steels D, E and F. These Steels correspond to industrial pure irons, and are out of the inventive chemical composition. As shown in Nos.5 and 6, the remarkable coarsening of the ferrite grains could not be expected in spite of the annealing at not lower than 1000° C. Further, the strain was introduced during transformation from an austenite to a ferrite, and desired properties were not therefore imparted. No.7 shows results when the annealing temperature was lower than the transformation point, and so good properties were not provided.

Table 1

$K_i^{n_d}$ s	Chemical Composition (wt%)									"X"
	C	Si	Mn	P	S	Sol. Al	N	O		
A	0.0007	0.01	0.01	0.0015	0.0009	1.39	0.0028	0.0015	No	
B	0.0009	0.01	0.01	0.0010	0.0008	1.10	0.0029	0.0014	No	
C	0.0010	0.01	0.01	0.0015	0.0010	0.98	0.0021	0.0017	No	
D	0.0025	0.01	0.01	0.0010	0.0006	0.001	0.0027	0.0360	885°C	
E	0.0010	0.01	0.11	0.0080	0.0062	0.001	0.0030	0.0200	890°C	
F	0.0030	0.01	0.09	0.0050	0.0021	0.001	0.0041	0.0088	885°C	
G	0.013	0.02	0.15	0.020	0.012	0.017	0.0035	0.002	—	

Note "X" : Transformation

Table 2

No	$K_{\text{fnd}}^{\text{I}}$	"Y" (°C)	μ_{max}	H _c (Oe)	B _{0.2} (G)	B _{0.5} (G)	B ₁ (G)	B _{2.5} (G)	"Z" (μm)	
1	A	1100	60500	0.27	10500	13300	14000	16000	3.0	Inv.
2	A	1000	23900	0.36	1800	11200	13700	16200	0.8	"
3	B	1100	56000	0.30	9500	12800	13800	16200	2.5	"
4	C	1050	37200	0.35	7100	12100	13800	16300	2.0	"
5	D	1100	12500	0.69	300	5400	12000	16400	0.2	Com.
6	E	1000	11000	0.74	400	3600	9400	16500	0.2	"
7	F	850	11500	0.80	200	1900	11500	16700	0.2	"
8	G	890	7290	0.7	—	—	—	—	—	" *

Comparative Example *: From Japanese Patent Publication

No.63-45443

Note "Y": Annealing temperature

"Z": Average ferrite crystal grain sizes

Inv.: Example of the invention,

Com.: Comparative Example

Example 2

Table 3 shows both chemical compositions of the inventive Examples and the comparative Examples. With respect to Steels I to U, steel ingots of 110 mm thickness were made from melts, and the ingots were hot rolled to 15 mm thickness by heating 1200°C. Steels I to S, W to Y, Z and b to d fall within the inventive chemical composition, while Steels T, U, V and a are comparative steels. Table 4 shows results of the DC magnetization properties measured and the ferrite grain sizes of the inventive steels and the comparative steels. In the annealings of the present Example, the heating - holding times were 1 to 3 hours,

and the cooling rates were around 100 ° C/hr to 500 ° C/hr.

In Table 4, Nos.10 to 13 varied the Mn content within the ranges specified by the invention.

Nos.23 to 26 observed influences of the sol.Al content, No.28 observed influences of the C content, and Nos.29 to 31 observed influences of the Si content.

5 Nos.14 to 16 added Ti. Also herein, the ferrite single phase were made by the Al addition, and further N was fixed by the Ti addition. Nos.14 to 16 show desirable properties. No.15 is a special example where Ti was added to a steel equivalent to No.22 in accordance with the invention, and N was sufficiently fixed by Ti addition so that a great improvement was observed in comparison with the comparative example of No.22.

10 No.21 is a comparative example where Ti was added more than the specified range of the invention, and the DC magnetization property is remarkably deteriorated.

No.22 is a comparative example where N addition was high and Ti was not added. Since a precipitation of AlN was stable, the ferrite grains were not fully coarsened in spite of the annealing, and a solute N content was high so that satisfied properties could not be realized. Nos.17 and 18 are examples where
15 Steels P and Q were annealed at 1000 ° C.

Each of Nos.10 to 18, Nos.24 to 26, No.27 and Nos.29 to 31 not only can accomplish the excellent DC magnetization property where the coercive force is not more than 0.4 Oe and the $B_{0.5}$ value is not less than 10000 G and by far satisfy the properties specified in JIS C 2504 SUYPO but also may be applied to as the magnetic shielding material for presenting magnetic field circumstances of a magnetic field level below the
20 earth magnetism.

Nos.19 and 20 investigated influences of Ti in relation with the N content and the C + N content, and the both had N > 0.005 wt% and C + N > 0.007 wt%, but No.20 obtained a desired properties due to Ti addition.

Each of the inventive examples show the desirous DC magnetization property, and has coarse ferrite
25 grain of not less than 0.5 mm.

As is seen from the above mentioned, the soft magnetic ferrous materials according to the invention have the excellent DC magnetization properties and may be easily magnetized even in weak magnetic fields, and those are useful as iron core materials of high functions or magnetic shielding material of high function.

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Table 3

K _{inds}	Chemical Composition (wt%)								
	C	Si	Mn	P	S	Sol. Al	N	O	Ti
I	0.0009	0.01	0.05	0.0007	0.0002	1.20	0.0015	0.0008	tr.
J	0.0007	0.01	0.11	0.0004	0.0002	1.19	0.0013	0.0011	tr.
K	0.0008	0.01	0.16	0.0003	0.0003	1.22	0.0012	0.0008	tr.
L	0.0008	0.01	0.36	0.0005	0.0003	1.25	0.0016	0.0010	tr.
M	0.0009	0.01	0.01	0.0015	0.0010	1.12	0.0021	0.0012	0.01
N	0.0015	0.01	0.02	0.0015	0.0008	1.16	0.0116	0.0023	0.06
O	0.0012	0.01	0.01	0.0015	0.0005	1.27	0.0007	0.0016	0.83
P	0.0023	0.01	0.01	0.0010	0.0003	0.73	0.0009	0.0020	tr.
Q	0.0022	0.01	0.01	0.0009	0.0002	1.17	0.0009	0.0022	tr.
R	0.0020	0.01	0.01	0.0011	0.0004	1.24	0.0070	0.0018	tr.
S	0.0022	0.01	0.01	0.0009	0.0005	1.25	0.0067	0.0025	0.03
T	0.0014	0.01	0.01	0.0011	0.0002	1.17	0.0008	0.0047	4.30
U	0.0018	0.01	0.02	0.0013	0.0004	1.13	0.0126	0.0016	tr.
V	0.0020	0.01	0.01	0.0008	0.0004	0.30	0.0013	0.0023	tr.
W	0.0017	0.01	0.01	0.0006	0.0003	0.96	0.0016	0.0016	tr.
X	0.0023	0.03	0.01	0.0005	0.0003	1.32	0.0008	0.0020	tr.
Y	0.0014	0.03	0.01	0.0005	0.0003	1.63	0.0011	0.0017	tr.
Z	0.0015	0.01	0.01	0.0006	0.0005	1.25	0.0036	0.0018	tr.
a	0.0080	0.01	0.01	0.0006	0.0005	1.12	0.0009	0.0008	tr.
b	0.0008	0.06	0.01	0.0006	0.0006	1.22	0.0016	0.0012	tr.
c	0.0008	0.17	0.01	0.0006	0.0006	1.23	0.0014	0.0010	tr.
d	0.0013	0.50	0.01	0.0006	0.0006	1.23	0.0014	0.0012	tr.

Table 4

No	Kinds	"V" (°C)	μ_{max}	H _c (Oe)	B _{0.5} (G)	B ₁ (G)	B ₂₅ (G)	"W" (mm)	
10	I	1100	69000	0.14	13700	14300	16500	3.5	Inv.
11	J	1100	64000	0.18	12800	13300	15900	2.5	"
12	K	1100	70000	0.14	13300	13800	16100	4.0	"
13	L	1000	47000	0.22	12200	13100	15900	3.0	"
14	M	1050	58200	0.20	13500	14300	16400	2.5	"
15	N	1100	46000	0.22	13600	14500	16500	1.0	"
16	O	1100	48200	0.18	12600	13900	15800	1.5	"
17	P	1000	64000	0.16	13800	14400	16600	3.0	"
18	Q	1000	67700	0.15	14000	14600	16600	3.5	"
19	R	1100	21000	0.47	10700	13800	16500	1.0	Com.
20	S	1100	62000	0.22	13100	14100	16400	2.0	Inv.
21	T	1100	1500	2.5	200	600	12100	0.3	Com.
22	U	1100	23000	0.50	11400	13900	16500	0.4	"
23	V	1000	22000	0.39	10400	13600	16500	0.3	"
24	W	1100	64800	0.19	14500	15100	17000	2.5	Inv.
25	X	1100	71400	0.16	13500	14100	16300	4.0	"
26	Y	1100	49000	0.19	13100	13400	16000	3.0	"
27	Z	1100	53000	0.27	13500	14300	16600	2.5	"
28	a	1100	19700	0.46	9700	13900	16600	0.4	Com.
29	b	1100	45000	0.25	13300	14000	16000	2.0	Inv.
30	c	1100	48000	0.21	13000	13500	16000	2.0	"
31	d	1100	48000	0.23	12600	12900	15500	1.5	"

Note "V": Annealing temperatures; "W": Average ferrite crystal grain sizes
 Inv.: Example of the invention; Com.: Comparative Example

INDUSTRIAL APPLICABILITY

The present invention may be applied to production of soft magnetic materials, for example, electromagnetic cores of magnetic shielding materials which require high DC magnetization properties.

Claims

1. A method of producing soft magnetic ferrous materials characterized by heating not lower than 700 °C but not higher than 1300 °C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.5 wt%, Mn: not more than 0.50 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol. Al: 0.5 to 2.0 wt%, N: not more than 0.005 wt%, oxygen: not more than 0.005 wt%, and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700 °C, and finally annealing at temperatures of 900 to 1300 °C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.
2. The method as defined in claim 1, wherein C + N is not more than 0.007 wt%.
3. A method of producing soft magnetic ferrous materials characterized by heating not lower than 700 °C but not higher than 1300 °C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.1 wt%, Mn: not more than 0.15 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol. Al: 0.5 to 2.0 wt%, N: not more than 0.005 wt%, oxygen: not more than 0.005 wt%, and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700 °C, and finally annealing at temperatures of 1000 to 1300 °C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.
4. The method as defined in claim 3, wherein C + N is not more than 0.007 wt%.
5. A method of producing soft magnetic ferrous materials characterized by heating not lower than 700 °C but not higher than 1300 °C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.5 wt%, Mn: not more than 0.50 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol. Al: 0.5 to 2.0 wt%, N: not more than 0.012 wt%, oxygen: not more than 0.005 wt%, Ti: 0.005 to 1.0 wt% and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700 °C, and finally annealing at temperatures of 900 to 1300 °C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.
6. The method as defined in claim 5, wherein C + N is not more than 0.014 wt%.
7. A method of producing soft magnetic ferrous materials characterized by heating not lower than 700 °C but not higher than 1300 °C a steel piece or a cast piece composed of C: not more than 0.004 wt%, Si: not more than 0.1 wt%, Mn: not more than 0.15 wt%, P: not more than 0.015 wt%, S: not more than 0.01 wt%, sol. Al: 0.5 to 2.0 wt%, N: not more than 0.012 wt%, oxygen: not more than 0.005 wt%, Ti: 0.005 to 1.0 wt% and the rest being Fe and unavoidable impurities, accomplishing a hot working at temperatures of not lower than 700 °C, and finally annealing at temperatures of 1000 to 1300 °C, thereby to obtain a soft magnetic ferrous material having a coercive force of not more than 0.4 Oe and the magnetic flux density of not less than 10000 G at the magnetic field of 0.5 Oe.
8. The method as defined in claim 7, wherein C + N is not more than 0.014 wt%.

INTERNATIONAL SEARCH REPORT

International Application No PCT/JP89/01231

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int. Cl ⁵ C21D8/12, C22C38/06		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
IPC	C21D8/12, C22C38/06	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	JP, A, 60-208417 (Sumitomo Metal Industries, Ltd.), 21 October 1985 (21. 10. 85), Page 2, lower right column, 17th line from the bottom to page 3, upper left column, line 14 (Family: none)	1 - 8
X	JP, A, 57-35626 (Nippon Steel Corporation), 26 February 1982 (26. 02. 82), Page 2, upper right column, line 19 to lower left column, 6th line from the bottom (Family: none)	1 - 8
¹⁰ Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
March 3, 1990 (03. 03. 90)	March 19, 1990 (19. 03. 90)	
International Searching Authority	Signature of Authorized Officer	
Japanese Patent Office		