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(54) **METHOD FOR PRODUCING FE-CO ALLOY ROD**

(57) Provided is a method for producing an Fe-Co alloy rod whereby superior magnetic properties can be reliably obtained. This method for producing an Fe-Co alloy rod comprises a heating/straightening step in which tensile stress is applied while heating a hot rolled material

of an Fe-Co alloy to a temperature of 500 to 900°C. The Fe-Co alloy comprising, by mass%: 95% or more of Fe + Co; 25 to 60% of Co; 1.70 to 2.10% of V; 0.01 to 0.15% of Mn; 0.008% or less of C; 0.01% or less of S; and inevitable impurities as the balance.

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Description

Technical Field

5 **[0001]** The present invention relates to a method for producing an Fe-Co alloy rod and an Fe-Co alloy rod.

Related Art

10 **[0002]** Fe-Co alloy rods, represented by Permendur (also referred to as Permendur), possess excellent magnetic properties and are used in various products such as sensors, cylindrical magnetic shields, electromagnetic valves, and magnetic cores. As a method for producing these Fe-Co alloy rods, for instance, Patent Document 1 describes that an ingot is heated to 1000°C to 1100°C, then hot worked into a billet of approximately $\phi 90$ mm, removing surface defects by lathe, and after reheating to 1000°C to 1100°C, then hot-rolled to produce a material (rod) of approximately $\phi 6$ to $\phi 9$ mm.

15 **[0003]** Moreover, in Patent Document 2, the applicant of the present invention proposes an Fe-Co-based alloy rod, which can stably obtain excellent magnetic properties and has 20% or more grains having a GOS (Grain Orientation Spread) value of 0.5° or more in area ratio; and a method for producing the Fe-Co-based alloy rod, which includes a heating and straightening step that applies tensile stress while heating the hot-rolled material to a temperature of 500 to 900°C.

Citation List

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Patent Literature

[0004]

25 Patent Document 1: Japanese Patent Application Laid-Open Publication No. H7-166239
Patent Document 2: International Publication No. WO 2021/182518

SUMMARY OF INVENTION

30 Technical Problem

[0005] With the advancement in performance of the aforementioned products, further improvement in magnetic properties is required for the materials. It is difficult to consistently obtain high magnetic properties using conventional production methods as described in Patent Document 1, and there remains room for further investigation. The Fe-Co alloy rod described in Patent Document 2 possesses excellent magnetic properties and is a highly useful invention. On the other hand, while rods that omit the solution treatment excel in strength, they may exhibit slightly lower magnetic properties compared to rods that have undergone solution treatment, leaving room for improvement. Therefore, an objective of the present invention is to provide a method for producing Fe-Co alloy rods capable of consistently obtaining excellent magnetic properties.

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Solution to Problem

[0006] The present invention has been made in consideration of the aforementioned issues.

45 **[0007]** In other words, an aspect of the present invention is a method for producing an Fe-Co alloy rod, which includes a heating and straightening step of applying tensile stress to a hot-rolled material of the Fe-Co alloy consisting of, in mass%, Fe+Co: 95% or more, Co: 25 to 60%, V: 1.70 to 2.10%, Mn: 0.01 to 0.15%, C: 0.008% or less, S: 0.01% or less, and remainder inevitable impurities, while heating the hot-rolled material of the Fe-Co alloy to a temperature of 500 to 900°C.

[0008] Preferably, no solution treatment is performed before performing the heating and straightening step.

50 Effects of Invention

[0009] According to the present invention, it is possible to obtain Fe-Co alloy rods capable of consistently achieving excellent magnetic properties.

55 DESCRIPTION OF THE EMBODIMENTS

[0010] The following describes embodiments of the present invention. First, the method for producing Fe-Co alloy rods of the present invention is described. The Fe-Co alloy rods of the present invention are straight rod-shaped rods including

those with circular (including elliptical) or rectangular cross-sectional shapes. Unless otherwise specified, the rods in this embodiment are round rods with a circular cross-sectional shape.

Composition of Hot-Rolled Material

[0011] First, the reasons for limiting the composition of the Fe-Co alloy specified in the present invention are described. Fe+Co: 95 mass% (hereinafter also referred to simply as %) or more, Co: 25 to 60%

[0012] In the present invention, an Fe-Co alloy with Fe+Co of 95% or more and Co of 25 to 60% is used. The preferable lower limit of Co is 40%. This enables the achievement of high magnetic flux density and high permeability. The preferable content of Fe+Co is 97% or more.

V: 1.70 to 2.10%

[0013] V is included at 1.70% or more in the present invention because if it is too low, it causes embrittlement and reduces workability in subsequent steps. On the other hand, if the amount of V is too high, it not only invites a decrease in magnetic flux density but also causes a large amount of fine V-based carbides to disperse in the matrix, leading to a decrease in excitation characteristics and iron loss characteristics. Therefore, the addition is limited to 2.10% or less in mass%. The preferable upper limit of V is 2.00%. Furthermore, to further improve magnetic properties, the preferable upper limit of V is 1.95%.

Mn: 0.01 to 0.15%

[0014] Mn is effective in improving excitation characteristics and iron loss characteristics by fixing the impurity element S as MnS through its addition. However, if it is excessive, it conversely causes a decrease in excitation characteristics and iron loss characteristics. In particular, excessive addition of Mn becomes a factor that lowers the γ phase precipitation temperature during magnetic annealing. Therefore, in the present invention, the addition is limited to 0.01 to 0.15% or less. The preferable upper limit of Mn is 0.10%.

C: 0.008% or less, S: 0.01% or less

[0015] C and S are impurity elements that hinder good excitation characteristics and iron loss characteristics. If C exceeds 0.005% or S remains above 0.01%, good excitation characteristics and iron loss characteristics cannot be consistently obtained. Therefore, in the present invention, C and S are limited to C: 0.008% or less and S: 0.01% or less. The preferable upper limit of C is 0.005%, and the preferable upper limit of S is 0.005%. Other inevitable impurity elements include, for instance, Si, P, and O, and the upper limit of each of them is preferable set as, for instance, 0.1%.

[0016] In the Fe-Co alloy of the present invention, the V and Mn that are actively added satisfy the above-mentioned ranges, and further, it is preferable that a total amount of V and Mn is in the range of 2.1% or less. This tends to suppress excessive coercivity and consistently obtain good excitation characteristics and iron loss characteristics. Moreover, as mentioned above, Mn fixes the impurity element S as MnS through its addition. Therefore, to obtain a more reliable effect of fixing the impurity S, it is preferable to set the Mn:S ratio in the range of 10 to 100.

[0017] In this embodiment, as an intermediate material for the Fe-Co alloy rod, a hot-rolled material may be obtained by applying hot-rolling to a billet obtained from an Fe-Co alloy steel ingot having the aforementioned components. Since an oxide layer is formed on this intermediate material by hot-rolling, a grinding step may be introduced to remove the oxide layer mechanically or chemically, for instance.

[0018] This hot-rolled material has, for instance, a shape corresponding to a "hot-rolled rod" of the Fe-Co alloy rod. Moreover, considering the workability in subsequent steps, the diameter may be set to 5 to 20 mm. Furthermore, for rods other than round rods, the circle equivalent diameter of the cross-section may be set to 5 to 20 mm.

<Solution Treatment Step>

[0019] In this embodiment, at least one solution treatment may be performed on the hot-rolled material before the heating and straightening step to be described later. It is preferable to perform this solution treatment because it can remove component segregation in the hot-rolled material, improve magnetic properties, and also improve workability. The heating temperature during this solution treatment tends to deteriorate workability if it is too low, and cause deterioration of magnetic properties if it is too high, so it is preferable to perform it at a temperature of 800 to 1050°C. A more preferable lower limit of temperature is 850°C. A more preferable upper limit of temperature is 950°C, and an even more preferable upper limit of temperature is 900°C. The heating time may also be set to 10 to 60 minutes. In the solution treatment step, rapid cooling is performed after heating to dissolve harmful precipitates without precipitating them and to suppress

ordering, thereby improving workability.

[0020] Moreover, even if the solution treatment step is omitted, the effects of the present invention may be obtained by adjusting the heating temperature in the heating and straightening step to be described later. Preferably, the solution treatment step is omitted (not performed). While the Fe-Co alloy composition in this embodiment described above has excellent magnetic properties, it tends to form coarse grains compared to conventional Fe-Co alloys, which may cause variations in strain applied to the material during the heating and straightening step, potentially leading to unstable magnetic properties. By omitting the solution treatment step, the structure of the hot-rolled material may be made into a uniform fine-grained rolled structure with less strain variation in the heating and straightening step, suppressing the formation of coarse grains in the Fe-Co alloy rod after magnetic annealing, and it is possible to consistently have coercivity equal to or higher than that of conventional solution-treated products and to enhance mechanical strength.

<Heating and straightening step>

[0021] In this embodiment, a heating and straightening step is performed on the aforementioned hot-rolled material, applying tensile stress while heating. At this time, if the hot-rolled material is in the shape of a "rod", this tensile stress is applied by pulling the hot-rolled rod in the length direction. This step allows for obtaining a rod with excellent magnetic properties and straightness while imparting residual strain to the hot-rolled material. The heating temperature at this time is set to 500 to 900°C. In the case where it is lower than 500°C, workability decreases, and there is a risk of the rod breaking when applying tensile stress. On the other hand, in the case where the heating temperature exceeds 900°C, it is not possible to impart desirable residual strain to the hot-rolled material. The preferable lower limit of the heating temperature in the heating and straightening step is 600°C, and more preferably 700°C. Moreover, the preferable upper limit of the heating temperature is 850°C, more preferably 830°C, and even more preferably 800°C. Furthermore, in the case where the aforementioned solution treatment step is omitted, the preferable lower limit of the heating temperature is 700°C, more preferably 730°C, and even more preferably 740°C.

[0022] In this heating and straightening step, heating means such as electric heating or induction heating may be used, but it is preferable to apply electric heating due to its advantages of easily aligning the easy magnetization axis of grains in the hot-rolled material in a certain direction, and rapidly (for instance, within 1 minute) and uniformly heating the material to a target temperature. Moreover, it is preferable to adjust the tensile load applied to the rod during the heating and straightening step to 4 to 90 kN in order to more reliably obtain the desired residual strain. Further, it is preferable to adjust the elongation to 3 to 10% of the total length before the heating and straightening step.

[0023] In this embodiment, centerless grinding using a centerless grinder, for instance, may be performed on the rod that has completed the heating and straightening step. This allows for removing the black skin from the surface layer of the rod and further improving the roundness and tolerance accuracy of the shape. In the present invention, since the straightness of the rod is improved by the heating and straightening step, centerless grinding may be performed on long rods with lengths of 1000 mm or more without cutting them.

[0024] The Fe-Co alloy rod obtained by the production method of the present invention described above has excellent magnetic properties (for instance, coercivity of 42 A/m or less, and magnetic flux density (B2000) of 2.2 or more at an applied magnetic field of 2000 A/m). Here, in the case where stable magnetic properties and high strength are desired to be achieved simultaneously, it is preferable to omit the aforementioned solution treatment step. In the case where further improvement in magnetic properties is desired, solution treatment may be performed. Furthermore, in the case where the solution treatment is omitted, the coercivity may be 42 A/m or less, and the 0.2% yield strength after magnetic annealing may be 200 MPa or more. A more preferable 0.2% yield strength is 210 MPa or more. This 0.2% yield strength may be measured based on the tensile testing method for metallic materials according to JIS Z2241. In the case where solution treatment is performed, the coercivity may be reduced to 35 A/m or less. The Fe-Co alloy rod obtained by the production method of the present invention preferably has a grain size number of 5.0 or more and 11.0 or less. This facilitates achieving high magnetic properties after magnetic annealing. In the case where the solution treatment step is omitted to achieve both stable magnetic properties and high strength, the preferable lower limit of the grain size number for the Fe-Co alloy rod is 7.0, and the preferable upper limit of the grain size number is 10.5. A more preferable lower limit of the grain size number is 7.5, and a more preferable upper limit of the grain size number is 10.0. In the case of performing solution treatment to further improve magnetic properties, the preferable lower limit of the grain size number is 5.5, and the preferable upper limit of the grain size number is 7.0. Moreover, the grain size number may be measured based on JIS G0551. Furthermore, measured may be performed on the cross-section perpendicular to the axis or the axial cross-section of the rod.

Implementation Example

(Implementation Example 1)

[0025] An Fe-Co alloy steel ingot having the composition shown in Table 1 was bloomed, and then hot-rolled to prepare a hot-rolled rod with a diameter of ϕ 11.5 mm.

<No. 1, No. 11>

[0026] After performing a solution treatment in which the aforementioned hot-rolled rod was heated to 850°C and then rapidly cooled, the hot-rolled rod was subjected to a heating and straightening step in which it was heated to a temperature of 750°C while being stretched in its length direction under a tensile load condition of 27 kN, so as to produce Fe-Co alloy rods as sample No. 1, which is an example of the present invention, and sample No. 2, which is a comparative example. Moreover, a total amount of inevitable impurities was approximately 0.1% or less.

<Samples No. 2, No. 12>

[0027] The Fe-Co alloy rods as sample No. 2, which is an example of the present invention, and sample No. 12, which is a comparative example, were produced by performing the heating and straightening step on the aforementioned hot-rolled rod without performing the solution treatment. The conditions for the heating and straightening step were the same as those for sample No. 1. Moreover, the total amount of inevitable impurities was approximately 0.1% or less.

[Table 1]

Sample No.	C	Si	Mn	P	S	Co	V	Remainder
1, 2	0.001	0.04	0.05	0.006	0.001	48.92	1.89	Fe and inevitable impurity
11	0.010	0.04	0.13	≤ 0.01	≤ 0.01	49.01	1.97	Fe and inevitable impurity
12	0.010	0.04	0.13	≤ 0.01	≤ 0.01	49.25	1.99	Fe and inevitable impurity

[0028] Subsequently, the grain size number, DC magnetic properties, and 0.2% yield strength of the samples from the examples of the present invention and the comparative examples were confirmed. The average crystal grain size was determined by observing ten fields of view of $500\ \mu\text{m} \times 350\ \mu\text{m}$ on the cross-section (cross-section perpendicular to the axis) using an Olympus optical microscope, and the grain size number was determined using the crystal grain size standard plate I in accordance with JIS G0551. The samples were observed on the longitudinal cross-section (axial cross-section passing through the center axis) of the aforementioned samples. For the DC magnetic properties, after collecting samples from the obtained rods, magnetic annealing was performed at 850°C for 3 hours, and the coercivity was measured using DC magnetization specific test equipment. In the examples of the present invention, in addition to the coercivity, the magnetic flux density (B2000) at an applied magnetic field of 2000 A/m was also measured. The 0.2% yield strength at room temperature was measured using a 1/2 scale JIS No. 4 test piece specified in JIS Z2241, and the 0.2% yield strength was measured based on the JIS Z2241 tensile test method for metallic materials. Table 2 shows the observation results.

[Table 2]

Sample No.	Grain Size Number	Coercivity (A/m)	Magnetic flux density B2000 (T)	0.2% yield strength (MPa)	Remark
1	6.0	32	2.24	180	Example of the present invention
2	8.0	37	2.26	214	Example of the present invention
11	8.0	39	-	191	Comparative example
12	11.0	43	-	213	Comparative example

[0029] From Table 2, it may be confirmed that sample No. 1, which is an example of the present invention that underwent solution treatment, has the smallest grain size number of 6.5 or less (largest grain diameter), and shows an excellent coercivity of 33 A/m or less. It also has a magnetic flux density (B2000) of 2.20 T or higher, which reproduces the high magnetic flux density characteristic of Permendur, confirming that it has excellent magnetic properties. Even in the Sample No. 2, which is an example of the present invention that omitted the solution treatment, the grain size number is 8.0 and the

coercivity is 38 A/m or less, which is lower than the coercivity of sample No. 11, a comparative example that underwent solution treatment. The magnetic flux density is also at the same level as sample No. 1 (2.20 T or higher), and 0.2% yield strength is 200 MPa or higher and the highest among the four samples. Therefore, it was confirmed that a good balance between favorable magnetic properties and high strength is achieved.

(Implementation Example 2)

[0030] Ten samples each were prepared under the same production conditions as sample No. 1 in Implementation Example 1 with solution treatment completed, and ten samples each were prepared under the same production conditions as sample No. 2 in Implementation Example 1 without solution treatment. After collecting samples, magnetic annealing was performed at 850°C for 3 hours, and the coercivity was measured using DC magnetization specific test equipment. When a coercivity of 42 A/m or less was deemed to be qualified, five out of ten samples with solution treatment completed were judged to be acceptable, whereas all ten samples without solution treatment were judged to be acceptable. This suggests that samples without solution treatment are capable of consistently exhibiting favorable magnetic properties.

Claims

1. A method for producing an Fe-Co alloy rod, comprising:
a heating and straightening step of applying tensile stress to a hot-rolled material of an Fe-Co alloy consisting of, in mass%, 95% or more of Fe+Co, 25 to 60% of Co, 1.70 to 2.10% of V, 0.01 to 0.15% of Mn, 0.008% or less of C, 0.01% or less of S, and remainder being inevitable impurities, while heating the hot-rolled material to a temperature of 500 to 900°C.
2. The method for producing the Fe-Co alloy rod according to claim 1, wherein no solution treatment is performed before performing the heating and straightening step.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2023/032124

A. CLASSIFICATION OF SUBJECT MATTER

C21D 8/12(2006.01)i; **C22C 30/00**(2006.01)i; **C22C 19/07**(2006.01)i; **C22C 38/00**(2006.01)i; **C22C 38/12**(2006.01)i;
C22F 1/00(2006.01)n; **C22F 1/10**(2006.01)i; **H01F 1/147**(2006.01)i
 FI: C22F1/10 J; C21D8/12 F; C22C19/07 C; C22C30/00; C22C38/00 303S; C22C38/12; H01F1/147; C22F1/00 624;
 C22F1/00 630A; C22F1/00 660C; C22F1/00 683; C22F1/00 684C; C22F1/00 685A; C22F1/00 691B; C22F1/00 691C;
 C22F1/00 691Z; C22F1/00 694B

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C21D8/12; C22C30/00; C22C19/07; C22C38/00; C22C38/12; C22F1/00; C22F1/10; H01F1/147

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2023
 Registered utility model specifications of Japan 1996-2023
 Published registered utility model applications of Japan 1994-2023

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2021/182518 A1 (HITACHI METALS, LTD.) 16 September 2021 (2021-09-16) claims, paragraphs [0008]-[0021], tables 1-4	1-2
A	JP 2004-515644 A (IMPHY UGINE PRECISION) 27 May 2004 (2004-05-27) entire text	1-2
A	US 2009/0184790 A1 (VACUUMSCHMELZE GMBH & CO. KG) 23 July 2009 (2009-07-23) entire text, all drawings	1-2

☐ Further documents are listed in the continuation of Box C.
 ☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

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WO 2021/182518 A1	16 September 2021	CN 115279926 A claims, paragraphs [0019]- [0050], tables 1-4	
JP 2004-515644 A	27 May 2004	US 2004/0099347 A1 entire text WO 2001/086665 A1 EP 1281182 A1 KR 10-0711188 B1 FR 2808806 A1 AU 6041201 A AT 465500 T ES 2342766 T3	
US 2009/0184790 A1	23 July 2009	(Family: none)	

Form PCT/ISA/210 (patent family annex) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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- WO 2021182518 A [0004]