(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 3 050 977 A1

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
03.08.2016 Bulletin 2016/31

(21) Application number: **14849656.5**

(22) Date of filing: **22.09.2014**

(51) Int Cl.:
C21D 6/00 *(2006.01)* ***H01F 1/153*** *(2006.01)*
H01F 41/02 *(2006.01)* ***C22C 38/00*** *(2006.01)*
C22C 45/02 *(2006.01)*

(86) International application number:
PCT/JP2014/075070

(87) International publication number:
WO 2015/046140 (02.04.2015 Gazette 2015/13)

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME

(30) Priority: **27.09.2013 JP 2013201030**

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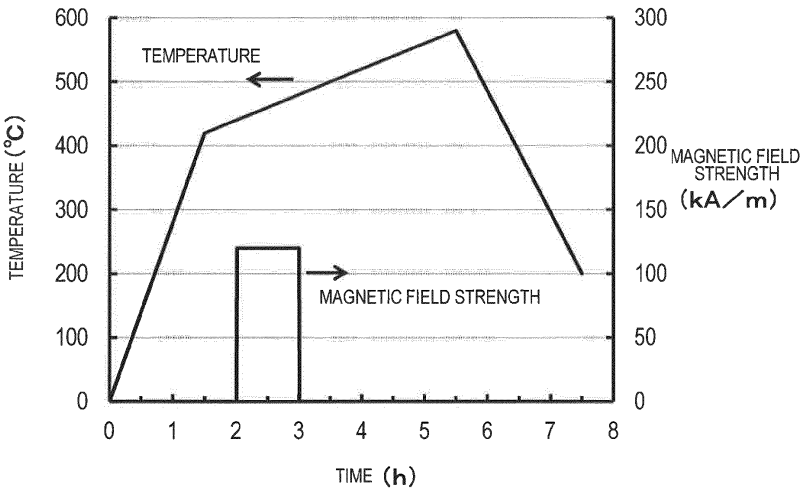
(54) METHOD FOR PRODUCING Fe-BASED NANO-CRYSTAL ALLOY, AND METHOD FOR PRODUCING Fe-BASED NANO-CRYSTAL ALLOY MAGNETIC CORE

(57) A method for producing an Fe-based nano-crystal alloy ribbon, includes a heat treatment step of heating a nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon. In the heat treatment step, a magnetic field is applied in a width direction of the alloy ribbon in a temperature range during a temperature-increasing period, the temperature range including at least a part of a temperature range from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature and not exceeding a temperature higher by 50°C than the crystallization start temperature, the crystallization start temperature being defined by a differential scanning calorimeter.

FIG.1



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Description

TECHNICAL FIELD

[0001] The present invention relates to a method for producing an Fe-based nano-crystal alloy and a method for producing a magnetic core formed of an Fe-based nano-crystal alloy in a wound or stacked state.

BACKGROUND ART

[0002] An Fe-based nano-crystal alloy has splendid soft magnetic characteristics that include both of a high saturation magnetic flux density and a high specific magnetic permeability μ, and therefore is used for a magnetic core of a common mode choke coil, a high frequency transformer and the like.

[0003] A representative composition system of such an Fe-based nano-crystal alloy is an Fe-Cu-Nb-Si-B system described in Patent Document 1.

[0004] An Fe-based nano-crystal alloy is produced as follows. A liquid-phase alloy heated to a temperature higher than, or equal to, a melting point thereof is rapidly solidified to obtain an amorphous alloy, and the amorphous alloy is heat-treated to be formed into nano-crystals. For rapidly solidifying the liquid-phase alloy, for example, a single roll method, which provides a high productivity, is used.

[0005] An Fe-based nano-crystal alloy has the magnetic characteristics thereof, such as the specific magnetic permeability μ, the squareness ratio and the like, changed in accordance with the temperature profile or the direction in which the magnetic field is applied, both at the time of heat treatment.

[0006] For example, Patent Document 2 proposes performing heat treatment while applying a magnetic field in a width direction of a ribbon (height direction of a magnetic core) in order to obtain an Fe-based nano-crystal alloy having an initial specific magnetic permeability of 70,000 or greater and a squareness ratio of 30% or less. Patent Document 2 describes various specific patterns of heat treatment, which may be roughly classified as follows: a pattern in which the ribbon is kept for a certain duration at the highest temperature reached during the heat treatment, while the magnetic field is applied; a pattern in which the ribbon is kept at a certain temperature for a certain duration during a procedure including a temperature-increasing process, when the ribbon is at the highest temperature reached and a cooling process, while the magnetic field is applied; and a pattern in which the ribbon is kept at a certain temperature for a certain duration during a procedure including when the ribbon is at highest temperature reached and a cooling process, while the magnetic field is applied.

CITATION LIST

PATENT LITERATURE

[0007]

Patent Document 1: Japanese Patent Publication for Opposition No. H04-4393

Patent Document 2: Japanese Laid-Open Patent Publication No. H07-278764

SUMMARY OF INVENTION

TECHNICAL PROBLEM

[0008] The heat treatment method disclosed in Patent Document 2 is considered to be effective as a method for decreasing the squareness ratio.

[0009] Recently, a high frequency band at or around 100 kHz tends to be used more for a common mode choke or the like. There is a strong demand for compact magnetic components usable in such a high frequency band. Namely, a nano-crystal alloy having a high specific magnetic permeability μ in the high frequency band is desired.

[0010] The present inventors performed various studies in order to obtain a high specific magnetic permeability μ in a high frequency band at or around 100 kHz, and as a result, have recognized that it is occasionally difficult to obtain a high specific magnetic permeability μ in the high frequency band by the heat treatment patterns described in Patent Documents 1 and 2.

[0011] The present invention made in light of the above situation has an object of providing a method for producing an Fe-based nano-crystal alloy and a method for producing an Fe-based nano-crystal alloy magnetic core, both easily providing a high specific magnetic permeability μ at or around a high frequency of 100 kHz.

SOLUTION TO PROBLEM

[0012] The present inventors have found that a high specific magnetic permeability μ in a high frequency band of, for example, 100 kHz is obtained by applying a magnetic field in a specific temperature range during a temperature-increasing period while an Fe-based amorphous alloy is nano-crystallized by heat treatment.

<1> Method for producing an Fe-based nano-crystal alloy

[0013] A method for producing an Fe-based nano-crystal alloy in an embodiment according to the present invention includes a heat treatment step of heating a nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon. In the heat treatment step, a magnetic field is applied in a width direction of the alloy ribbon in a temperature range during

a temperature-increasing period, the temperature range including at least a part of a temperature range from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature and not exceeding a temperature higher by 50°C than the crystallization start temperature, the crystallization start temperature being defined by a differential scanning calorimeter. Namely, the magnetic field is applied selectively in the temperature range during the temperature-increasing period.

[0014] In an embodiment, the magnetic field is applied at a strength of 50 kA/m or greater and 300 kA/m or less in the width direction of the alloy ribbon.

[0015] In an embodiment, the magnetic field is not applied when a highest temperature was reached in the heat treatment step.

[0016] In an embodiment, a method for producing an Fe-based nano-crystal alloy ribbon includes a step of preparing a nano-crystallizable Fe-based amorphous alloy ribbon, a step of heating the nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon, and a step of applying a magnetic field to the Fe-based amorphous alloy ribbon in the heat treatment step. In the step of applying the magnetic field, a magnetic field of a predetermined strength (e.g., 50 kA/m) or greater is applied in a width direction of the alloy ribbon in at least a part of a temperature range during a temperature-increasing period in the heat treatment step, the temperature range being from a temperature lower by 50°C than a crystallization start temperature, indicated by a differential scanning calorimeter, to a temperature higher by 20°C than the crystallization start temperature, and the magnetic field of the predetermined strength or greater is not applied in a part of the temperature-increasing period. Typically, the magnetic field of the predetermined strength or greater is not applied in a temperature-increasing period higher than a temperature higher by 50°C than the crystallization start temperature. It is not necessary to apply the magnetic field of the predetermined strength or greater in a temperature-increasing period lower than a temperature lower by 50°C than the crystallization start temperature.

<2> Method for producing an Fe-based nano-crystal alloy magnetic core

[0017] A method for producing an Fe-based nano-crystal alloy magnetic core in an embodiment according to the present invention includes a heat treatment step of heating a nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon after the nano-crystallizable Fe-based amorphous alloy ribbon is wound or stacked. Thus, a magnetic core including an Fe-based nano-crystal alloy ribbon in a wound or stacked state is produced. In the heat treatment step, a magnetic field is applied in a height direction of the magnetic core in a temperature range during a temperature-increasing period, the temperature range including at least a part of a temperature range from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature and not exceeding a temperature higher by 50°C than the crystallization start temperature, the crystallization start temperature being defined by a differential scanning calorimeter. Namely, the magnetic field is applied selectively in the temperature range during the temperature-increasing period.

[0018] In an embodiment, the magnetic field is applied at a strength of 50 kA/m or greater and 300 kA/m or less in the height direction of the magnetic core.

[0019] In an embodiment, the Fe-based nano-crystal alloy ribbon has a thickness of 15 μm or less and a width of 250 mm or less.

ADVANTAGEOUS EFFECTS OF INVENTION

[0020] According to a method for producing an Fe-based nano-crystal alloy or a method for producing an Fe-based nano-crystal alloy magnetic core in an embodiment of the present invention, a high specific magnetic permeability μ at a high frequency of or around, for example, 100 kHz is easily realized. Therefore, the Fe-based nano-crystal alloy or the Fe-based nano-crystal alloy magnetic core are preferably usable for a common mode choke or the like, for which high frequency characteristics are important.

BRIEF DESCRIPTION OF DRAWINGS

[0021]

[FIG. 1] FIG. 1 is a graph illustrating a heat treatment profile and a magnetic field application profile in example 1 according to the present invention.

[FIG. 2] FIG. 2 is a graph illustrating a heat treatment profile and a magnetic field application profile in example 2 according to the present invention.

[FIG. **3**] FIG. **3** is a graph illustrating a heat treatment profile and a magnetic field application profile in example 3 according to the present invention.

[FIG. **4**] FIG. **4** is a graph illustrating a heat treatment profile and a magnetic field application profile in example 4 according to the present invention.

[FIG. **5**] FIG. **5** is a graph illustrating a heat treatment profile and a magnetic field application profile (no magnetic field) in comparative example 1.

[FIG. **6**] FIG. **6** is a graph illustrating a heat treatment profile and a magnetic field application profile in comparative example 2.

[FIG. **7**] FIG. **7** is a graph illustrating a heat treatment profile and a magnetic field application profile in comparative example 3.

DESCRIPTION OF EMBODIMENTS

[0022] Hereinafter, embodiments of the present invention will be described in detail.

[0023] One feature of the method for producing an Fe-based nano-crystal alloy and an Fe-based nano-crystal alloy magnetic core in an embodiment according to the present invention is the following. During heat treatment performed while an amorphous alloy is supplied with a magnetic field to obtain an Fe-based nano-crystal alloy, the magnetic field is selectively applied in a specific temperature range during a temperature-increasing period, unlike in the conventional art. The magnetic field is applied in a width direction of a ribbon, namely, in a height direction of a magnetic core.

[0024] This will be described specifically. The above-described specific temperature range during the temperature-increasing period in the heat treatment step includes at least a part of a temperature range that is from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature but does not exceed a temperature higher by 50°C than the crystallization start temperature. In such a specific temperature range, the magnetic field is selectively applied in the width direction of the alloy ribbon that is being heat-treated. The crystallization start temperature is defined by a differential scanning calorimeter.

[0025] As can be seen, in the embodiment according to the present invention, the magnetic field is not applied, for example, at or around the highest temperature reached or during a cooling period after the highest temperature reached. The magnetic field is applied in the above-described period during the temperature-increasing period. However, the present inventors have confirmed that a relatively weak magnetic field (e.g., less than 50 kA/m) does not substantially decrease the specific magnetic permeability μ at a frequency of 100 kHz even if being applied at or around the highest temperature reached in the heat treatment. Therefore, in the embodiment according to the present invention, a relatively weak magnetic field may be applied temporarily or continuously in an optional period in the heat treatment step. In the embodiment according to the present invention, application of a weak magnetic field of less than 50 kA/m may not be considered as application of a magnetic field. Unless otherwise specified, the magnetic field described in the following description is of a level that can influence the magnetic characteristics of a nano-crystal alloy (typically, 50 kA/m or greater and 300 kA/m or less).

[0026] According to the results of experiments performed by the present inventors, when a magnetic field is applied at a highest temperature reached that exceeds a temperature higher by 50°C than the crystallization start temperature, typically defined by a differential scanning calorimeter, a large induced magnetic anisotropy is provided. When this occurs, the specific magnetic permeability μ in a region including a low frequency region and a high frequency region is generally decreased, and the specific magnetic permeability μ at the target frequency, namely, 100 kHz is low.

[0027] By contrast, in the case where a magnetic field is applied at or around the crystallization start temperature confirmed by the differential scanning calorimeter, a weak induced magnetic anisotropy is provided. The magnetic permeability at the target frequency, namely, 100 kHz has been confirmed to show a tendency of improving instead of decreasing. In the case where the magnetic field is applied at or around the crystallization start temperature, the specific magnetic permeability μ fluctuates little despite the fluctuation in the strength of the applied magnetic field or fluctuation in the temperature region in which the magnetic field is applied. It has been found that in this case, the specific magnetic permeability μ is easily adjustable at the target frequency, namely, 100 kHz.

[0028] A reason why the specific magnetic permeability μ is made easily adjustable especially in a high frequency band by applying a magnetic field during the temperature-increasing period as described above is presumed as follows, although the reason may not be completely accurate.

[0029] An alloy of an amorphous structure before being heat-treated has a Curie temperature lower than the crystallization start temperature. By contrast, when being nano-crystallized, the alloy has a Curie temperature significantly exceeding the crystallization start temperature. Namely, it is presumed that when a magnetic field is applied during crystallization, the magnetic domain of the alloy is secured along with the crystallization, and thus substantially the same effect as that provided when the alloy is cooled from a temperature higher than, or equal to, the Curie temperature is provided.

[0030] However, during the temperature-increasing period in which the structure is being kept changed, an induced magnetic anisotropy as strong as that provided when the alloy is cooled from the Curie temperature or the vicinity thereof is not provided. Based on this, it is presumed that the degree of the induced magnetic anisotropy of the alloy has been made easily controllable.

[0031] As described above, in the heat treatment step in the embodiment according to the present invention, a magnetic field is applied in at least a part of the temperature-increasing period, more specifically, in at least a part of a temperature range from a temperature lower by 50°C by the crystallization start temperature, which is defined by a differential scanning calorimeter, to a temperature higher by 20°C than the crystallization start temperature. In addition, in this embodiment, a magnetic field is applied in a temperature-increasing period in which the temperature does not exceed a temperature higher by 50°C than the crystallization start temperature.

[0032] In the case where a magnetic field is applied only in a temperature region that is lower than the temperature lower by 50°C than the crystallization start temperature, crystallization does not occur substantially. The

application of a magnetic field at such a temperature is performed while an amorphous state having a low Curie temperature is kept. Therefore, the above-described effect is not provided. By contrast, in the case where a magnetic field is applied only in a temperature region that is higher than the temperature higher by 20°C than the crystallization start temperature, the temperature approaches the Curie temperature of a nano-crystal alloy. Therefore, an excessively large induced magnetic anisotropy is provided, which makes it difficult to adjust the specific magnetic permeability μ.

[0033] More preferably, the temperature range in which the magnetic field is to be applied includes at least a part of a temperature range from a temperature lower by 20°C than the crystallization start temperature, which is defined by the differential scanning calorimeter, to a temperature higher by 10°C than the crystallization start temperature.

[0034] In the case where a magnetic field is kept applied in a temperature range including a low temperature region and a significantly high temperature region during the temperature-increasing period, an excessively large induced magnetic anisotropy is provided. In this case also, the specific magnetic permeability μ is made difficult to be adjusted. Therefore, in this embodiment, the upper limit of the temperature at which the magnetic field is to be applied is set to a temperature higher by 50°C than the crystallization start temperature. More preferably, the upper limit of the temperature at which the magnetic field is to be applied is a temperature higher by 40°C than the crystallization start temperature.

[0035] As can be seen from the above, in the embodiment according to the present invention, effective application of a magnetic field at a predetermined strength or greater (e.g., 50 kA/m or greater) is performed in a part of the temperature-increasing period, but is not performed throughout the temperature-increasing period. Namely, the temperature-increasing period includes a period in which effective application of a magnetic field is not performed. In this manner, effective application of a magnetic field is selectively performed in a temperature range around the crystallization start temperature; for example, effective application of a magnetic field is not performed in a temperature region lower by a temperature exceeding 50°C than the crystallization start temperature or in a temperature region higher by a temperature exceeding 50°C than the crystallization start temperature (at or around the highest temperature reached). Such a method efficiently provides a nano-crystal alloy having an appropriate induced magnetic anisotropy.

[0036] In this specification, the term "temperature-increasing period" indicates a period before the highest temperature is reached. The "temperature-increasing period" may be a period in which the temperature is being increased, a period in which the temperature is being decreased, or a period in which the temperature is kept constant, as long as being before the highest temperature is reached.

[0037] In the embodiment according to the present invention, the crystallization start temperature is defined by a differential scanning calorimeter. It is difficult to accurately measure the true crystallization start temperature, and it is effective to identify the crystallization start temperature by differential scanning calorimeter (DSC). A temperature at which an exothermic reaction caused by start of nano-crystallization is detected while the temperature is increased is defined as the "crystallization start temperature". According to the present invention, the measurement by use of a differential scanning calorimeter is performed under the condition that the temperature is increased by 10°C per minute.

[0038] In the embodiment according to the present invention, the heat treatment temperature is preferably controlled such that the temperature distribution in an actual heat treatment furnace is $\pm$5°C or less in consideration of the capacity of the heat treatment furnace and the amount of heat generation caused by crystallization of an amorphous ribbon that is being heat-treated. Such a manner of control allows the magnetic characteristics of the post-heat treatment alloy to be stabilized.

[0039] In the embodiment according to the present invention, the strength of the magnetic field to be applied is preferably 50 kA/m or greater and 300 kA/m or less. When the magnetic field applied is too weak, it is difficult to provide an induced magnetic anisotropy under the actual work conditions. When the magnetic field applied is too strong, an excessively large induced magnetic anisotropy tends to be provided.

[0040] A more preferable range of the strength of the magnetic field to be applied is 60 kA/m or greater and 240 kA/m or less.

[0041] Regarding the time duration in which the magnetic field is to be applied, there is no specific limitation as long as the temperature range is as described above. The time duration is practically about 1 to about 180 minutes.

[0042] A nano-crystallizable Fe-based amorphous alloy usable in the embodiment according to the present invention has a composition represented by, for example, general formula: $(Fe_{1-a}M_a)_{100-x-y-z}$-$\alpha$-$\beta$-$\gamma Cu_xSi_y$-$B_zM'\alpha M''\beta X\gamma$ (atomic %) (in the formula, M is Co and/or Ni; M' is at least one element selected from the group consisting of Nb, Mo, Ta, Ti, Zr, Hf, V, Cr, Mn and W; M" is at least one element selected from the group consisting of Al, platinum group elements, Sc, rare earth elements, Zn, Sn and Re; X is at least one element selected from the group consisting of C, Ge, P, Ga, Sb, In, Be and As; and a, x, y, z, α, β and γ respectively fulfill $0 \le a \le 0.5$, $0.1 \le x \le 3$, $0 \le y \le 30$, $0 \le z \le 25$, $5 \le y + z \le 30$, $0 \le \alpha \le 20$, $0 \le \beta \le 20$, and $0 \le \gamma \le 20$).

[0043] An alloy having the above-described composition is melted at a temperature higher than, or equal to, a melting point thereof, and rapidly solidified by a single roll method. As a result, a lengthy amorphous alloy ribbon (thin strip) is obtained.

[0044] The amorphous alloy ribbon preferably has a

thickness of 10 to 30 μm. When the thickness is less than 10 μm, the ribbon has an insufficient mechanical strength and is easily ruptured while being handled. When the thickness exceeds 30 μm, a stable amorphous state is not easily obtained. In the case where the amorphous alloy ribbon is nano-crystallized and used for a high frequency use as a magnetic core, an eddy current is generated to the ribbon. The loss caused by the eddy current is larger as the ribbon is thicker. Therefore, the thickness of the ribbon is more preferably 10 to 20 μm.

[0045] The specific magnetic permeability μ at or around 100 kHz has a larger value as the ribbon is thinner. Therefore, the thickness of the ribbon is still more preferably 15 μm or less.

[0046] The amorphous alloy ribbon preferably has a width of 10 mm or greater in consideration of a practical shape of the magnetic core. Slitting a wider alloy ribbon reduces the cost. Therefore, it is preferable that the alloy ribbon is wide on the stage after rapid cooling. For being produced stably, the alloy ribbon preferably has a width of 250 mm or less. For being produced more stably, the alloy ribbon more preferably has a width of 70 mm or less.

[0047] The heat treatment for nano-crystallization is preferably performed in an inert gas such as nitrogen or the like. The highest temperature to be reached is preferably set to 550 to 600°C. A highest temperature reached that is lower than 550°C or higher than 600°C is not preferable because such a temperature increases the magnetic strain. It is not necessary to set the time duration in which the alloy is kept at the highest temperature reached. The alloy may be nano-crystallized even if being kept at the highest temperature reached for 0 minutes (even if not being kept at the highest temperature reached). In consideration of the heat capacity of the entire alloy to be heat-treated and the stability of the characteristics of the alloy, the alloy may be kept at the highest temperature reached for a time duration of greater than 0 minutes to 3 hours or less.

[0048] A temperature profile in the heat treatment may be, for example, as follows: from room temperature to a temperature at which the nano-crystallization is started or in the vicinity of such a temperature, the temperature is increased relatively rapidly at an increasing rate of 2 to 4°C/min. ; and from a temperature lower by 50°C than the temperature at which the nano-crystallization is started to the highest temperature reached, the temperature is increased at a low rate of 0.2 to 1°C/min. Such a temperature profile allows the nano-crystallization to be caused efficiently and stably. In the cooling process after the nano-crystallization, the alloy is preferably cooled at a cooling rate of 2 to 5°C/min. in a temperature region from the highest temperature reached to 200°C. Usually, the alloy may be retrieved to the air after being cooled down to 100°C or lower.

[0049] For producing a magnetic core in the embodiment according to the present invention, the heat treatment step may be performed as follows. The nano-crystallizable Fe-based amorphous alloy ribbon is wound or stacked, and then is heated to a crystallization temperature region and cooled. In a process of heating the alloy ribbon to the crystallization temperature region (temperature-increasing period), the magnetic field is applied as described above. The magnetic field may be applied in the height direction of the magnetic core, so that a desired induced magnetic anisotropy is provided.

(Example 1)

[0050] A molten alloy member having a composition, in atomic %, of 1% of Cu, 3% of Nb, 15.5% of Si, 6.5% of B and the remaining part of Fe and unavoidable impurities was rapidly cooled by a single roll method to obtain an Fe-based amorphous alloy ribbon having a width of 50 mm and a thickness of 13 μm. The Fe-based amorphous alloy ribbon was slit to have a width of 3 mm and then wound into a shape having an outer diameter of 20 mm and an inner dimeter of 10 mm. Thus, ten toroidal magnetic cores were produced. The crystallization start temperature of this alloy measured by differential scanning calorimetry (DSC) was 500°C.

[0051] The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. 1. The magnetic field was continuously applied throughout the temperature range of 440 to 480°C during the temperature-increasing period (temperature range from the temperature lower by 60°C than the crystallization start temperature to the temperature lower by 20°C than the crystallization start temperature). The magnetic field was applied in a width direction of the alloy ribbon, namely, a height direction of the magnetic cores. The magnetic field was set to have a strength of 120 kA/m. The highest temperature reached during the heat treatment was set to be 580°C.

[0052] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 27,000 to 30,000.

[0053] The measurement was performed by use of HP4194A produced by Agilent Technologies under the conditions of an oscillation level of 0.5 V and an average of 16. An insulated covered wire was caused to pierce a central part of each of the toroidal magnetic cores and connected with an input/output terminal for the measurement.

(Comparative example 1)

[0054] Ten toroidal magnetic cores were produced in a similar manner by use of an Fe-based amorphous alloy ribbon having substantially the same composition and size as those in example 1. The produced magnetic cores were heat-treated by the temperature profile in example 1 shown in FIG. 1 with no magnetic field application (with no magnetic field) as shown in FIG. 5.

[0055] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz

in the range of 20,000 to 24,000.

[0056] A comparison of comparative example 1 with no magnetic field application and example 1 against each other confirms that when a magnetic field is applied in the temperature range defined by the present invention, even if the temperature range is lower than the crystallization start temperature defined by DSC, the specific magnetic permeability μ at 100 kHz is clearly improved.

(Example 2)

[0057] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy ribbon as used in example 1. The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. 2. Only the temperature range in which the magnetic field was applied was different from that in example 1 (FIG. 1), and the other conditions were substantially the same as those in example 1. The magnetic field was applied in the temperature range of 480 to 520°C (temperature range from the temperature lower by 20°C than the crystallization start temperature to the temperature higher by 20°C than the crystallization start temperature).

[0058] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 31,000 to 32,000.

[0059] The specific magnetic permeability μ at 100 kHz obtained in example 2 is higher than that in example 1. This indicates that application of a magnetic field performed in a temperature range including the crystallization start temperature defined by DSC can further increase the specific magnetic permeability μ at 100 kHz even if the strength of the magnetic field is the same.

(Example 3)

[0060] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy ribbon as used in example 1. The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. 3. Only the strength of the magnetic field was different from that in example 2 (FIG. **2**), and the other conditions were substantially the same as those in example 2. The magnetic field was applied at a strength of 60 kA/m while the temperature was increased.

[0061] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 28,000 to 30,000.

(Example 4)

[0062] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy as used in example 1. The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. 4. Only the strength of the magnetic field was different from that in example 2 (FIG. 2), and the other conditions were substantially the same as those in example 2. The magnetic field was applied at a strength of 240 kA/m while the temperature was increased.

[0063] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 27,000 to 29,000.

[0064] In examples 2 through 4, only the strength of the magnetic field is significantly different. As compared with comparative example 1 with no magnetic field application, the specific magnetic permeability μ at 100 kHz in each of examples 2 through 4 is confirmed to be significantly higher.

(Comparative example 2)

[0065] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy ribbon as used in example 1. The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. **6.** In comparative example 2, the strength of the magnetic field and the time duration of magnetic application were substantially the same as those in examples 1 and 2 (FIG. **1** and FIG. **2**), but the magnetic field was applied while the temperature was raised from 560°C to the highest temperature reached of 580°C and then was decreased. The magnetic field was started to be applied at the temperature higher by 60°C than the crystallization start temperature.

[0066] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 24,000 to 25,000.

[0067] In comparative example 2, the specific magnetic permeability μ at 100 kHz was higher by merely 4, 000 than that in comparative example 1 with no magnetic field application. Comparative example 1 and comparative example 2 were compared against each other regarding the specific magnetic permeability μ at a frequency of 10 kHz. The specific magnetic permeability μ was about 80,000 in comparative example 1 and was about 35,000 in comparative example 2. The specific magnetic permeability μ was higher in comparative example 1. This is presumed to have occurred for the following reason: when a magnetic field is applied in a temperature region higher by a temperature exceeding 50°C than the crystallization start temperature, an excessively large magnetic anisotropy is provided and thus the specific magnetic permeability μ at 100 kHz is low.

(Comparative example 3)

[0068] Ten toroidal magnetic cores were produced in

a similar manner by use of substantially the same Fe-based amorphous alloy ribbon as used in example 1. The produced magnetic cores were heat-treated and supplied with a magnetic field by a temperature profile and a magnetic field application profile shown in FIG. **7.** The magnetic field was applied throughout the heat treatment step. The magnetic field applied had a strength of 290 kA/m.

[0069] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 14,000 to 15,000.

(Example 5)

[0070] A molten alloy member having a composition, in atomic %, of 1% of Cu, 2.5% of Nb, 13.5% of Si, 7.2% of B and the remaining part of Fe and unavoidable impurities was rapidly cooled by a single roll method to obtain an Fe-based amorphous alloy ribbon having a width of 60 mm and a thickness of 18 μm. The Fe-based amorphous alloy ribbon was slit to have a width of 3 mm and then wound into a shape having an outer diameter of 20 mm and an inner dimeter of 10 mm. Thus, ten toroidal magnetic cores were produced. The crystallization start temperature of this alloy was measured to be 480°C.

[0071] The produced magnetic cores were heat-treated by the heat treatment profile shown in FIG. **2.** The alloy ribbon was kept at 580°C. The magnetic field was applied in the temperature range of 480 to 520°C while the temperature was increased (temperature range from the crystallization start temperature to the temperature higher by 40°C than the crystallization start temperature). The magnetic field was applied in a width direction of the alloy ribbon, namely, a height direction of the magnetic cores. The magnetic field was set to have a strength of 120 kA/m.

[0072] The ten post-heat treatment magnetic cores (alloy) were evaluated. The specific magnetic permeability μ at 100 kHz was in the range of 19,000 to 22,000.

(Comparative example 4)

[0073] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy ribbon as used in example 5. The produced magnetic cores were heat-treated by a temperature profile and a magnetic field application profile shown in FIG. **6** with no magnetic field application (with no magnetic field) .

[0074] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 17,000 to 18,000.

[0075] A comparison of example 5 and comparative example 4 with no magnetic field application against each other confirms that application of a magnetic field in the temperature range around the crystallization start temperature clearly improves the specific magnetic permeability μ at 100 kHz.

(Example 6)

[0076] A molten alloy member having a composition, in atomic %, of 5% of Ni, 0.8% of Cu, 2.8% of Nb, 11% of Si, 9. 8% of B and the remaining part of Fe and unavoidable impurities was rapidly cooled by a single roll method to obtain an Fe-based amorphous alloy ribbon having a width of 50 mm and a thickness of 13 μm. The Fe-based amorphous alloy ribbon was slit to have a width of 3 mm and then wound into a shape having an outer diameter of 20 mm and an inner dimeter of 10 mm. Thus, ten toroidal magnetic cores were produced. The crystallization start temperature of this alloy was measure to be 480°C.

[0077] The produced magnetic cores were heat-treated by the heat treatment profile shown in FIG. **2.** The alloy ribbon was kept at 580°C. The magnetic field was applied in the temperature range of 480 to 520°C while the temperature was increased (temperature range from the crystallization start temperature to the temperature higher by 40°C than the crystallization start temperature). The magnetic field was applied in a width direction of the alloy ribbon, namely, a height direction of the magnetic cores. The magnetic field was set to have a strength of 120 kA/m.

[0078] The ten post-heat treatment magnetic cores (alloy) were evaluated. The specific magnetic permeability μ at 100 kHz was in the range of 15,000 to 17,000.

(Comparative example 5)

[0079] Ten toroidal magnetic cores were produced in a similar manner by use of substantially the same Fe-based amorphous alloy as used in example 6. The produced magnetic cores were heat-treated by a temperature profile and a magnetic field application profile shown in FIG. 6 with no magnetic field application (with no magnetic field) .

[0080] The ten post-heat treatment magnetic cores (alloy) had a specific magnetic permeability μ at 100 kHz in the range of 9,000 to 12,000.

[0081] A comparison of example 6 and comparative example 5 with no magnetic field application against each other confirms that application of a magnetic field in the temperature range around the crystallization start temperature clearly improves the specific magnetic permeability μ at 100 kHz.

(Example 7)

[0082] A molten alloy member having substantially the same alloy composition as that in example 1 (crystallization start temperature: 500°C) was rapidly cooled by a single roll method to obtain an Fe-based amorphous alloy ribbon having a width of 50 mm and a thickness of 18 μm. The Fe-based amorphous alloy ribbon was slit to have a width of 15 mm and then wound into a shape having an outer diameter of 31 mm and an inner dimeter

of 21 mm. Thus, four toroidal magnetic cores were produced.

[0083] The produced magnetic cores were heat-treated by the heat treatment profile shown in FIG. 2 as in example 2. The magnetic field was applied in the temperature range of 480 to 520°C while the temperature was increased. The magnetic field was applied in a width direction of the alloy ribbon, namely, a height direction of the magnetic cores. The magnetic field was set to have a strength of 120 kA/m.

[0084] The four post-heat treatment magnetic cores (alloy) were evaluated. The specific magnetic permeability μ at 100 kHz was in the range of 28,000 to 29,000.

[0085] A comparison of example 2 and example 7 against each other confirms that the specific magnetic permeability μ at 100 kHz is slightly higher in example 2 in which the thickness of the Fe-based amorphous alloy ribbon is 15 μm or less than in example 7 in which the thickness of the Fe-based amorphous alloy ribbon exceeds 15 μm.

INDUSTRIAL APPLICABILITY

[0086] The method for producing an Fe-based nano-crystal alloy in the embodiment according to the present invention is applicable to production of a magnetic core of a common mode choke coil, a high frequency transformer and the like.

Claims

1. A method for producing an Fe-based nano-crystal alloy, comprising:

   a heat treatment step of heating a nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon;
   wherein in the heat treatment step, a magnetic field is applied in a width direction of the alloy ribbon in a temperature range during a temperature-increasing period, the temperature range including at least a part of a temperature range from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature and not exceeding a temperature higher by 50°C than the crystallization start temperature, the crystallization start temperature being defined by a differential scanning calorimeter.

2. The method for producing an Fe-based nano-crystal alloy according to claim 1, wherein the magnetic field is applied at a strength of 50 kA/m or greater and 300 kA/m or less in the width direction of the alloy ribbon.

3. The method for producing an Fe-based nano-crystal alloy according to claim 1 or 2, wherein the magnetic field is not applied when a highest temperature was reached in the heat treatment step.

4. A method for producing an Fe-based nano-crystal alloy magnetic core formed of an Fe-based nano-crystal alloy ribbon in a wound or stacked state, comprising:

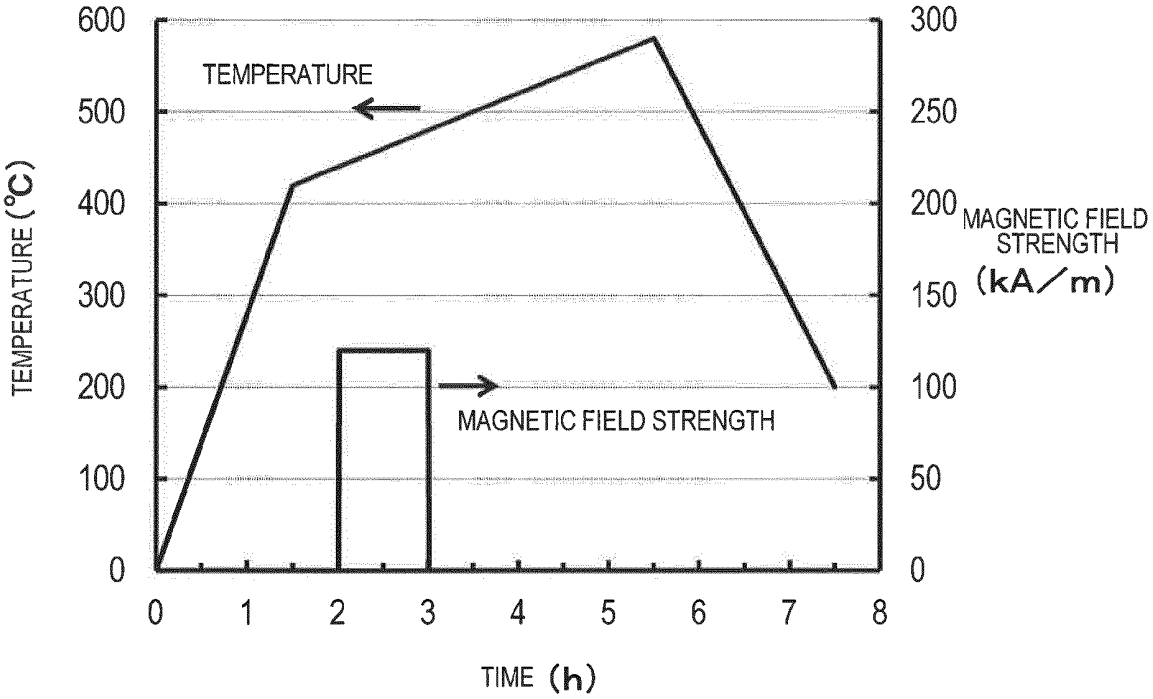
   a heat treatment step of heating a nano-crystallizable Fe-based amorphous alloy ribbon to a crystallization temperature region and cooling the nano-crystallizable Fe-based amorphous alloy ribbon after the nano-crystallizable Fe-based amorphous alloy ribbon is wound or stacked;
   wherein in the heat treatment step, a magnetic field is applied in a height direction of the magnetic core in a temperature range during a temperature-increasing period, the temperature range including at least a part of a temperature range from a temperature lower by 50°C than a crystallization start temperature to a temperature higher by 20°C than the crystallization start temperature and not exceeding a temperature higher by 50°C than the crystallization start temperature, the crystallization start temperature being defined by a differential scanning calorimeter.

5. The method for producing an Fe-based nano-crystal alloy magnetic core according to claim 4, wherein the magnetic field is applied at a strength of 50 kA/m or greater and 300 kA/m or less in the height direction of the magnetic core.

6. The method for producing an Fe-based nano-crystal alloy magnetic core according to claim 4 or 5, wherein the Fe-based nano-crystal alloy ribbon has a thickness of 15 μm or less and a width of 250 mm or less.

7. The method for producing an Fe-based nano-crystal alloy magnetic core according to any one of claims 4 through 6, wherein the magnetic field is not applied when a highest temperature is reached in the heat treatment step.

FIG.1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TEMPERATURE
MAGNETIC FIELD STRENGTH
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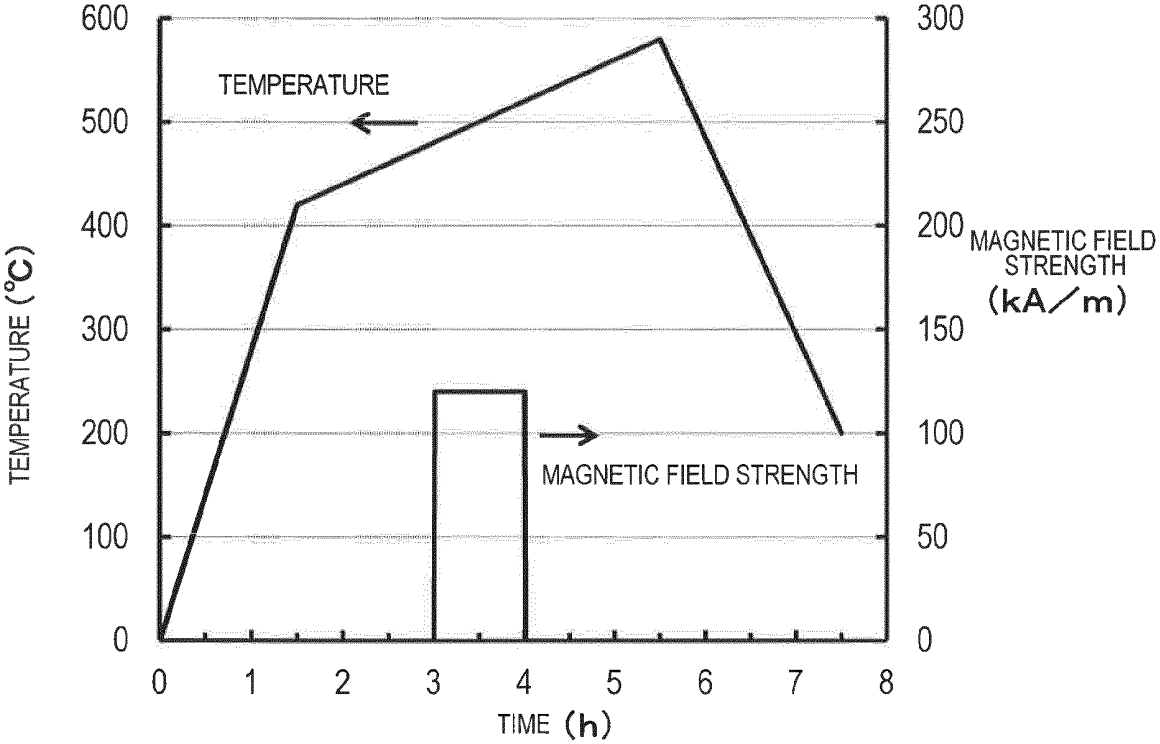
TIME (h)

FIG.2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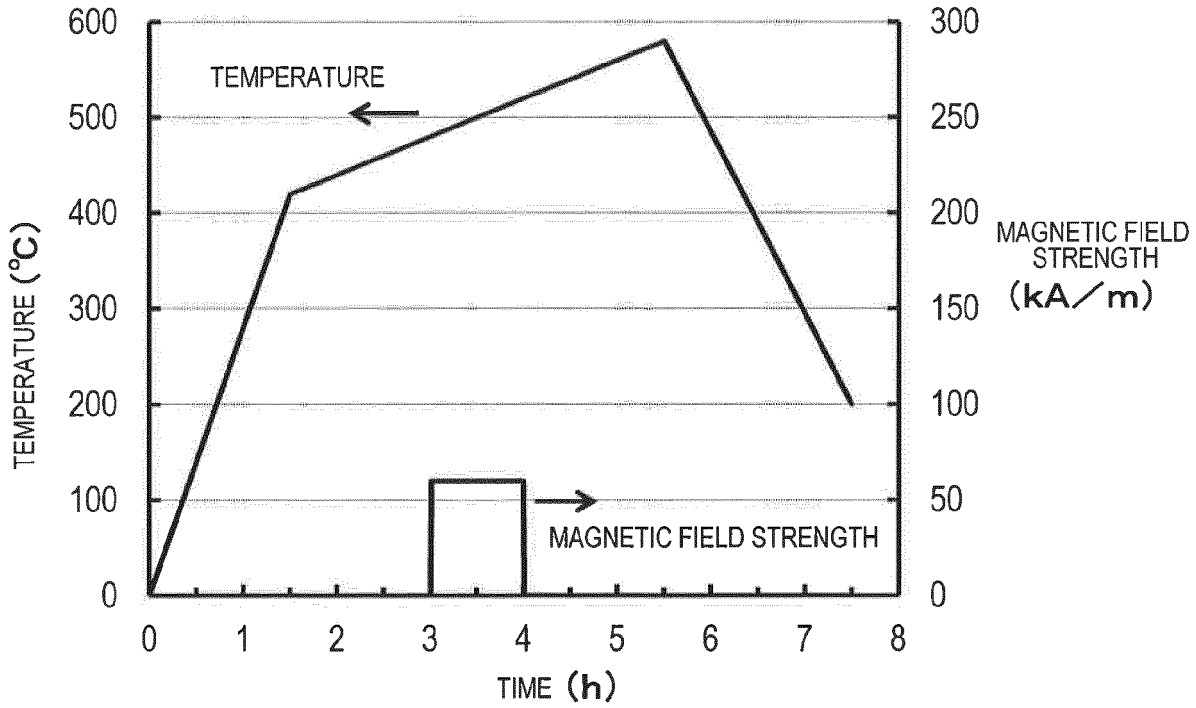
TEMPERATURE
MAGNETIC FIELD STRENGTH
0 1 2 3 4 5 6 7 8
TIME (h)

FIG.3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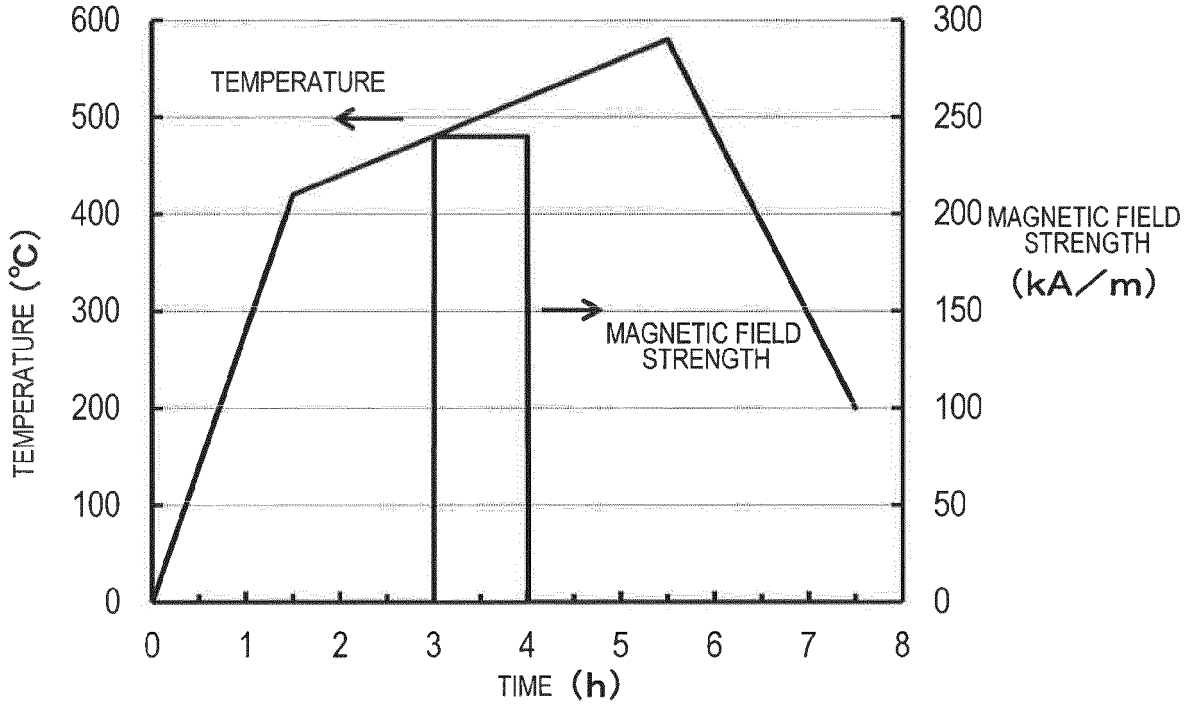
TIME (h)

FIG.4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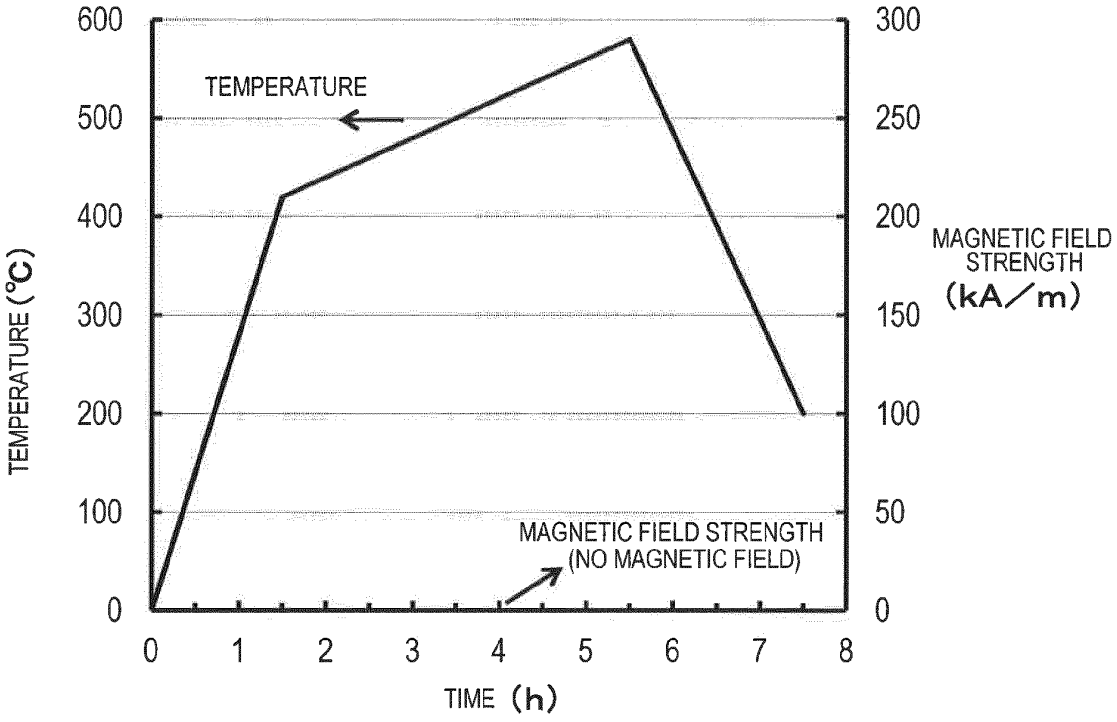
TIME (h)

FIG.5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TEMPERATURE
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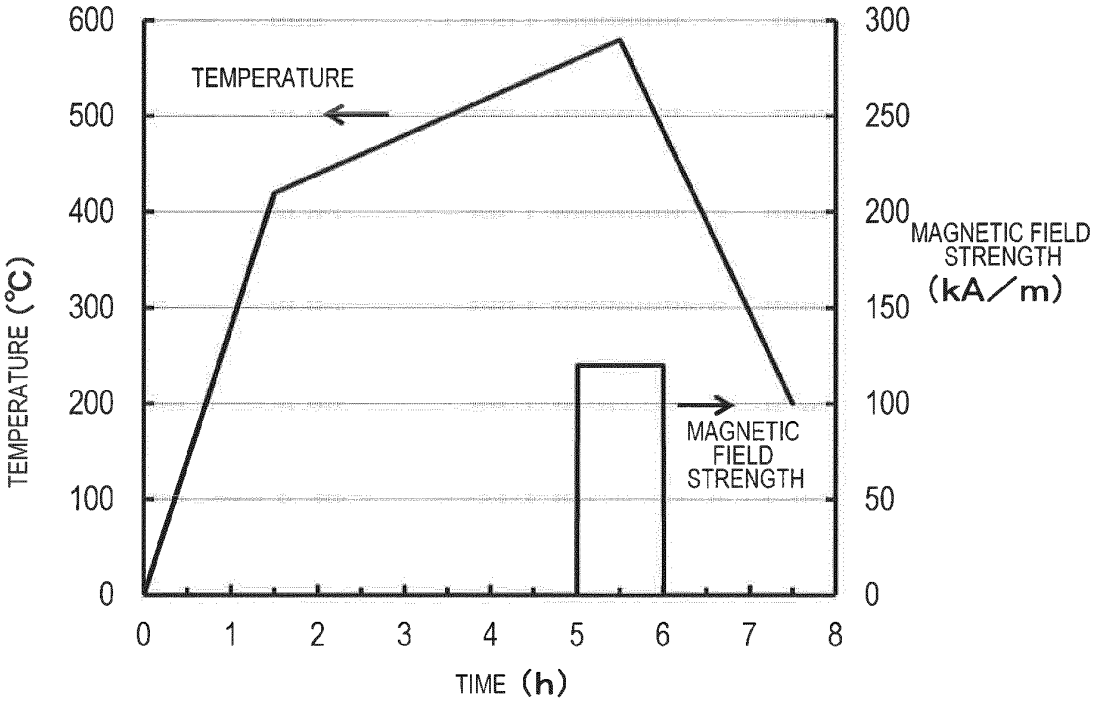
TIME (h)

FIG.6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TEMPERATURE
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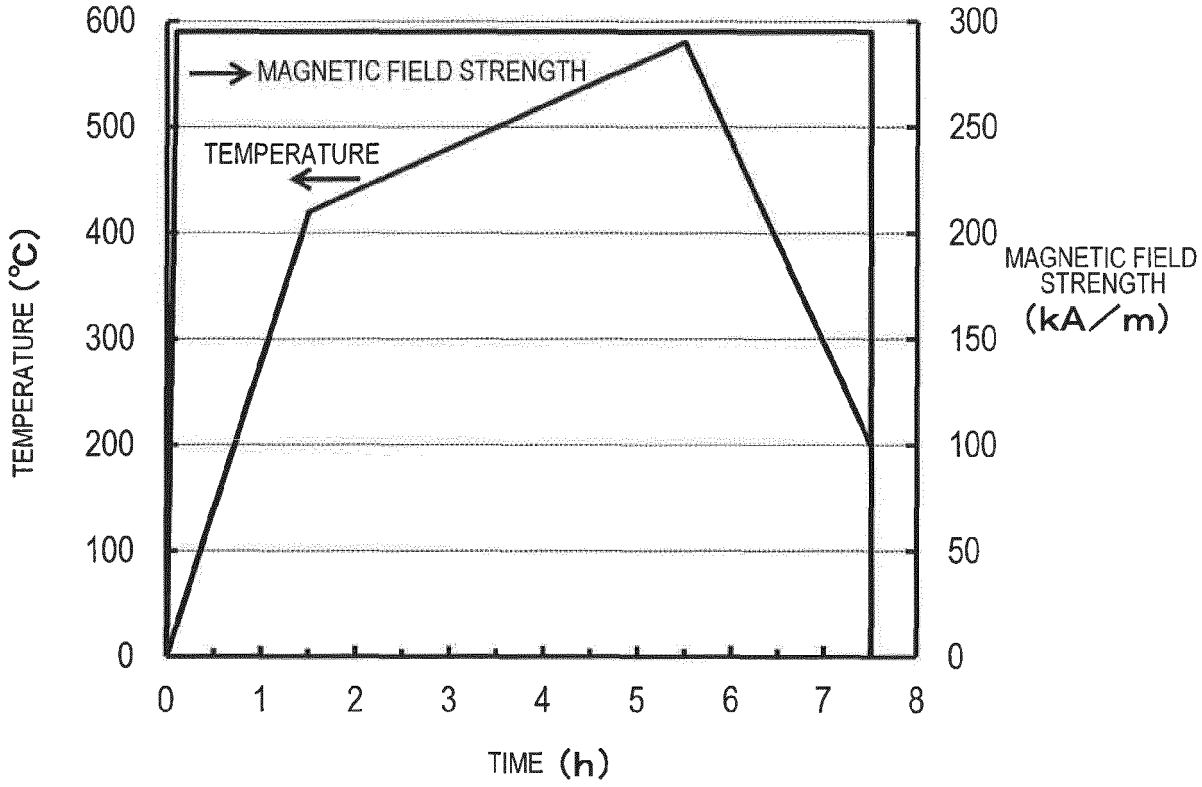
TIME (h)

FIG. 7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0 1 2 3 4 5 6 7 8
TIME (h)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2014/075070

A. CLASSIFICATION OF SUBJECT MATTER
C21D6/00(2006.01)i, *H01F1/153*(2006.01)i, *H01F41/02*(2006.01)i, *C22C38/00*(2006.01)n, *C22C45/02*(2006.01)n

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)
C21D6/00, H01F1/153, H01F41/02, C22C38/00, C22C45/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2014
Kokai Jitsuyo Shinan Koho 1971-2014 Toroku Jitsuyo Shinan Koho 1994-2014

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 A	JP 64-79342 A (Hitachi Metals, Ltd.), 24 March 1989 (24.03.1989), claims; page 19, lower right column, lines 14 to 15; page 25, lower right column, line 19 to page 26, upper right column, line 14; page 28, upper right column, line 7 to lower right column, line 4; fig. 33(e) & US 4881989 A & US 5160379 A & EP 271657 A2 & CA 1323219 A & KR 10-1991-0003977 B1	1,2,4-6 3,7
Y A	JP 2005-187917 A (Hitachi Metals, Ltd.), 14 July 2005 (14.07.2005), claims; 0007, 0017; fig. 1 (Family: none)	1,2,4,5 3,6,7

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

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
08 December, 2014 (08.12.14)	22 December, 2014 (22.12.14)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2014/075070

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	JP 5-202452 A (Sumitomo Metal Industries, Ltd.), 10 August 1993 (10.08.1993), entire text; all drawings (Family: none)	1-7
A	JP 2-77105 A (Hitachi Metals, Ltd.), 16 March 1990 (16.03.1990), entire text; all drawings & EP 299498 A1 & DE 3884491 T2 & CA 1341105 C & KR 10-1991-0002375 B1	1-7

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REFERENCES CITED IN THE DESCRIPTION

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- JP H044393 B **[0007]**
- JP H07278764 B **[0007]**