

# (11) EP 3 088 106 A1

(12)

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

(43) Date of publication: 02.11.2016 Bulletin 2016/44

(21) Application number: 14873470.0

(22) Date of filing: 26.11.2014

(51) Int Cl.: B22F 3/24<sup>(2006.01)</sup>

B22F 5/00 (2006.01)

(86) International application number: PCT/JP2014/081250

(87) International publication number: WO 2015/098407 (02.07.2015 Gazette 2015/26)

(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: 26.12.2013 JP 2013269343 04.09.2014 JP 2014180181 11.11.2014 JP 2014229131

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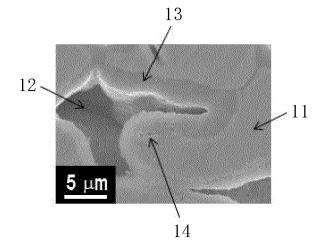
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# (54) MACHINE COMPONENT USING POWDER COMPACT AND METHOD FOR PRODUCING SAME

(57) A machine part having a radial crushing strength of more than 100 MPa is manufactured by: press forming raw material powder containing, as a main raw material, metal powder capable of forming an oxide film, to thereby

provide a green compact; and forming the oxide film between particles of the metal powder forming the green compact through steam treatment.

# FIG. 1A



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# Description

Technical Field

- [0001] The present invention relates to a machine part using a green compact and a method of manufacturing the same. More specifically, the present invention relates to a machine part obtained by increasing strength of a green compact, which is obtained by forming metal powder with a uniaxial working press or the like, without sintering, and to a method of manufacturing the same.
- 10 Background Art

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**[0002]** In the field of powder metallurgy, a product has hitherto been generally obtained by mixing raw material powders including metal powder, and compacting and forming the mixed powders, followed by sintering in a furnace at a high temperature of more than 800°C. A product directly after forming the metal powder is hereinafter referred to as "green compact" and is distinguished from a sintered compact obtained by further performing a sintering step (see JIS Z 2500: 2000).

**[0003]** According to JIS Z 2500: 2000, the powder metallurgy refers to a metallurgy technology category involving production of the metal powder and manufacturing of a product through forming of the metal powder and a sintering step. The powder metallurgy is a technology different from casting and forging, and the product is generally manufactured by the following steps.

- (1) Mixing of powders serving as raw materials, such as metal powder, lubricant powder, and graphite powder
- (2) Compacting and forming with a pressure press or the like
- (3) Sintering at a temperature equal to or lower than a melting point
- (4) Correction (sizing)
- (5) Post-processing, such as heat treatment or oil impregnation

(as required)

- [0004] Of those steps, (3) the sintering step generally includes treatment in a high temperature region of 800°C or more in the case of an iron-based material, and the cost of this step accounts for from 1/4 to 1/2 of the entire manufacturing cost. Further, a green compact expands or shrinks through the sintering step at high temperature, and hence (4) the correction step is indispensable in order to keep the dimensions and accuracy of the product within target dimensions and target accuracy.
- [0005] Through the sintering step, metal particles are fused with each other, that is, necking occurs, resulting in an increase in strength. However, when sufficient strength is secured through treatment at lower temperature, the manufacturing cost can be reduced. Besides, a dimensional change can be suppressed, and hence also the correction step can be omitted.

**[0006]** As a method of increasing the strength of a green compact other than the sintering, there have hitherto been investigations as described below.

**[0007]** In Patent Literature 1, there is a disclosure of a method of strengthening a green compact in the powder metallurgy. Specifically, the method includes compression molding metal powder having added thereto metal soap as a lubricant for forming, and then heating the resultant green compact at a temperature equal to or higher than the melting point of the metal soap and equal to or lower than the dewaxing temperature of the metal soap, to thereby significantly increase the mechanical strength of the green compact after cooling. The mechanism of this method is presumed as follows: the metal soap in pores in the green compact melts through the heat treatment to form a continuous layer and then solidifies, to thereby increase the strength of the green compact by virtue of the density of the layer (see Scope of Claim, lines 10-12 in the second column, and lines 22-25 in the third column of Patent Literature 1).

**[0008]** In Patent Literature 2, there is a disclosure that an iron-based "sintered" part is manufactured by subjecting a green compact to steam blackening treatment without sintering to bond particles of the green compact to each other. The mechanism of this technology is as follows: the entire surface of the green compact is covered with an oxide film through the steam blackening treatment, and hence an object in which particles on its surface are bonded and fixed to each other and thus predetermined strength is achieved in its entirety is obtained (lines 8-11 in the lower left column on page 2 of Patent Literature 2).

Citation List

[0009]

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Patent Literature 1: JP 61-011282 B2 Patent Literature 2: JP 63-072803 A Patent Literature 3: JP 04-198403 A

# 5 Summary of Invention

**Technical Problem** 

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**[0010]** An object of the technology disclosed in Patent Literature 1 is just to prevent chipping and breakage of the green compact during its conveyance to a sintering furnace from a forming press or the like. The green compact does not have strength as a product as it is. Therefore, as a matter of course, there is no suggestion of omission of the sintering step. The green compact cannot ultimately secure enough strength for its use as a product without a subsequent sintering step at high temperature. Besides, the number of steps is increased by one as compared to a general sintered product owing to the treatment before sintering, which contrarily causes an increase in cost.

**[0011]** The technology disclosed in Patent Literature 2 involves subjecting the green compact to the steam blackening treatment to bond the particles to each other through the oxide film and impart corrosion resistance. However, the disclosure "having strength and durability to some extent" (lines 7-8 in the upper right column on page 2) is entirely unclear as to what level of strength is actually obtained. In short, the technology disclosed in Patent Literature 2 is intended to "provide an inexpensive part which is easily manufactured for some applications not requiring very high strength among applications of a magnetic material part" (lines 10-12 in the upper left column on page 2). The applications of the part are limited to the applications not requiring high strength, such as a soft magnetic material part, which is given as a specific example. In actuality, there is no detailed disclosure of the material and density of the green compact, the conditions of the steam treatment, and the like, which are considered important for increasing the strength of the green compact.

**[0012]** An object of the present invention is to provide a machine part having strength comparable to that of a sintered product manufactured by the related art at lower cost by adopting, instead of the sintering step, another treatment at lower temperature.

**[0013]** Herein, a part having a radial crushing strength according to "Sintered metal bearing-Determination of radial crushing strength" of JIS Z 2507 of 100 MPa or more has strength enough to withstand the use as a machine part. In addition, according to "Powder metallurgy-Vocabulary" of JIS Z 2500, the "dimensional change" is defined as an increase or decrease in dimensions of a green compact to be generated through sintering, but herein means an increase or decrease in dimensions of the green compact to be generated not through a sintering step but through steam treatment.

# Solution to Problem

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**[0014]** As means for achieving the above-mentioned object, the inventors of the present invention have focused attention on steam treatment. In general, the steam treatment is also called homogenization treatment, and involves allowing an iron-based sintered material (material after sintering) and steam to react with each other while heating the iron-based sintered material at from about 500 °C to about 560 °C in an oxidizing atmosphere, to thereby form a film formed mainly of triiron tetraoxide ( $Fe_3O_4$ ) on a surface. The thickness of the film is said to be from about 3  $\mu$ m to about 7  $\mu$ m. There are few examples of applying the steam treatment to a general steel material, but the steam treatment has traditionally been widely utilized as inexpensive surface treatment for a sintered metal. The treatment has three main purposes of rust prevention, improvement in wear resistance, and sealing. In addition to the foregoing, the treatment is considered effective also for improvement in surface hardness and machinability (for example, see Patent Literature 3). **[0015]** The present invention is intended to clarify a method of obtaining a green compact having high strength by optimizing the material and density of the green compact, the conditions of the steam treatment, and the like. Herein, the "high strength" does not refer to strength at such a level that the chipping resistance of the green compact is improved or strength of a soft magnetic material part, and corresponds to such a level that the green compact can withstand the use as a machine element part, such as a sintered oil-impregnated bearing. The "high strength" specifically refers to a radial crushing strength of 100 MPa or more.

[0016] A related-art sintered compact is increased in strength through fusion (necking) between particles by compacting and forming raw material powder through uniaxial pressing or the like, followed by heating at a high temperature equal to or lower than a melting point (in the case of an iron-based material, from  $800^{\circ}$ C to  $1,300^{\circ}$ C). Meanwhile, in the present invention, a green compact having a specific density is allowed to react with steam at a high temperature of from  $400^{\circ}$ C to  $550^{\circ}$ C in an oxidizing atmosphere, to thereby form an oxide film on the surface of iron powder. An oxide is formed mainly of triiron tetraoxide (Fe $_3$ O $_4$ ) when iron-based powder is used as raw material powder. The oxide film to be formed between particles of the raw material powder plays the role of the necking between the particles of the powder instead, with the result that the strength of the green compact is increased.

[0017] The treatment temperature is low as compared to the general sintering step, and hence a dimensional change is small ( $\pm 0.1\%$  or less before and after the treatment). Therefore, a sizing step, which has hitherto been required for dimensional correction after sintering, can be omitted. In addition, a product and a mold for compacting and forming are designed with ease. Further, (electric or heat) energy required for the treatment can be reduced by virtue of the low treatment temperature. Besides, the number of treatment steps can be reduced, and the manufacturing process of the product can be shortened and the cost of the product can be reduced.

**[0018]** The steam treatment is atmosphere treatment, and hence is applicable irrespective of the shape and the size of dimensions of a product to be formed. In addition, the present invention is applicable to any material capable of forming an oxide film. Therefore, it is considered that the present invention is applicable not only to the iron-based material, but also to any metal material having a high ionization tendency, such as aluminum, magnesium, or chromium. While an atomizing method, a reduction method, a stamping method, a carbonyl method, and the like are known as a production method for the raw material powder, the present invention is applicable irrespective of the production method for the raw material powder.

**[0019]** Further, while uniaxial press forming is given as a typical example of a compacting method for the green compact, the present invention is applicable as long as a metal base material capable of forming an oxide film is exposed. Specifically, the present invention is also applicable to, for example, a green compact subjected to forming with a multiaxial CNC press or injection molding (MIM), or a green compact further subjected to cold isostatic press (CIP) forming, and a compacting and forming method is not limited as long as the powder is packed.

[0020] In general, lubricant powder, such as metal soap or amide wax, is mixed at the time of compacting and forming in order to secure lubrication between powder to be formed and a mold and between the respective powders, and the lubricant powder remains in the green compact. In the related-art methods, the lubricant powder is decomposed because the green compact is retained at high temperature in the subsequent sintering step, and hence is not contained in a product after sintering. However, when the present invention is applied, a lubricant component may remain depending on the density of the green compact, the treatment temperature, and a retention time. Therefore, it is desired to adopt such a method that a degreasing step of decomposing and removing the lubricant component is provided in advance prior to the steam treatment and the steam treatment is successively performed after the degreasing step. However, it has been found that an increase in strength can be achieved even when the steam treatment is performed with the lubricant contained without performing the degreasing step.

[0021] In general, a sintered part having a higher density has higher strength. Meanwhile, when a green density is too high, steam cannot penetrate an inside of the green compact, and the formation of the oxide film is limited to only a surface layer of the green compact. Therefore, such case is not preferred while the strength is increased. However, when the green density is too low, there are risks in that chipping or breakage occurs at the time of handling (a rattler value is large), and the oxide film is not formed between the particles owing to an excessively long distance between the particles. For the above-mentioned reasons, it is desired to set the green density to fall within a range of from 5.0 g/cm³ to 7.6 g/cm³, preferably from 5.3 g/cm³ to 7.2 g/cm³, more preferably from 6.0 g/cm³ or more and less than 7.0 g/cm³. The green density is measured by a dimension measurement method.

[0022] The surface of the machine part described above is covered with an iron oxide film, and hence has high surface hardness and excellent wear resistance as compared to a sintered compact obtained by subjecting a green compact having the same composition to general sintering treatment. In addition, according to investigations made by the inventors of the present invention, it has been revealed that, when the machine part is a part configured to slide with respect to a mating member through the intermediation of an oil (for example, a plain bearing, a gear, a cam, or the like to be used in an oil-lubricated environment), the machine part has an excellent initial running-in characteristic as compared to the sintered compact. The cause for this is not clearly understood, but a possible cause is as follows: as shown and illustrated in FIGS. 1, on the surface of the machine part (a green compact according to Example 8 described below), pores 12 in the order of several tens of micrometers remain between iron powders 11, and moreover, micropores 14 in the order of several tens of nanometers are formed in an inside of an iron oxide film 13 having a thickness of about several micrometers, and hence an oil impregnated in the micropores 14 contributes to the initial running-in characteristic.

Advantageous Effects of Invention

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**[0023]** According to the present invention, the following effects are obtained.

**[0024]** By allowing steam at high temperature to act on the green compact in an oxidizing atmosphere in a significantly low temperature region (e.g., a region of from 400°C to 550°C in the case of an iron-based material) as compared to a sintering step, particles are bonded to each other (the oxide film is formed), and hence high strength can be achieved. The "high strength" as used herein specifically means a radial crushing strength of 100 MPa or more. The machine part having a radial crushing strength of 100 MPa or more has sufficiently high strength as compared to the related art (Patent Literatures 1 and 2), and has strength enough to withstand practical use also as a machine part. For example, a machine element part which can serve as an alternative to a related-art sintered oil-impregnated bearing can be provided.

**[0025]** In addition, a dimensional change is small as compared to the case of sintering at high temperature by virtue of low treatment temperature. Therefore, the subsequent correction (sizing) step can be omitted. For the same reason, the manufacturing process can be shortened and the cost can be reduced. For example, the dimensional change before and after the steam treatment is suppressed to less than  $\pm 0.1\%$  with respect to the green compact. As a result, a product and a mold for compacting and forming are designed with ease.

**[0026]** Further, the present invention is applicable irrespective of the shape and dimensions of the green compact. The green compact is covered with the oxide film after the steam treatment, and hence anti-rust treatment is not required. When an additive that is altered or decomposed at a temperature of more than 500°C, for example, a material exhibiting slidability or lubricity is added depending on the treatment temperature, high functionalization of the product is achieved.

**Brief Description of Drawings** 

# [0027]

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- FIG. 1A is a photograph (SEM image) of a cross section of Example of the present invention.
- FIG. 1B is a view for schematically illustrating FIG. 1A.
- FIG. 2A is a front view of a friction and wear tester.
- FIG. 2B is a side view of the friction and wear tester.
- FIG. 3 is a graph for showing a convergence value of a friction coefficient in a friction and wear test (in presence of external oil feeding).
- FIG. 4 is a graph for showing transition of the friction coefficient in the friction and wear test (in presence of external oil feeding).
- FIG. 5 is a graph for showing a specific wear amount in the friction and wear test (in presence of external oil feeding).
- FIG. 6 is a graph for showing a convergence value of a friction coefficient in a friction and wear test (in absence of external oil feeding).
- FIG. 7 is a graph for showing transition of the friction coefficient in the friction and wear test (in absence of external oil feeding).
- FIG. 8 is a graph for showing a specific wear amount in the friction and wear test (in absence of external oil feeding).
- 30 Description of Embodiments
  - [0028] An embodiment of the present invention is described below with reference to specific Examples.
  - **[0029]** Test pieces of Examples 1 to 13 and Comparative Examples 1 to 10 were each produced by using reduced iron powder as base material metal powder, electrolytic copper powder as a second metal component, and an amide wax-based powder lubricant as a lubricant for compacting and forming, and subjected to various tests. The test pieces each have a ring shape having dimensions described below.

Inner diameter:  $\phi$  12 mm Outer diameter:  $\phi$  20 mm

Length: 7 mm

Examples 1 to 5

**[0030]** 2 wt% of the electrolytic copper powder and 0.7 wt% of the lubricant were added to the reduced iron powder, and the mixture was loaded into a mold made of alloy tool steel SKD 11 and subjected to uniaxial press forming at various forming pressures. Thus, five cylindrical green compacts having different green densities (g/cm³) were obtained. After that, a lubricant component in each green compact was subjected to degreasing treatment at 350 °C for 90 minutes, and then steam treatment was performed at 500°C for 40 minutes. Thus, the test pieces of Examples 1 to 5 were obtained. The green densities (g/cm³) in Examples 1 to 5 were as described below.

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Example 1: 5.3

Example 2: 6.0

Example 3: 6.5

Example 4: 7.0

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# Examples 6 to 9

**[0031]** In order to investigate the influence of a difference in treatment temperature of the steam treatment, green compacts each having a green density of 6.5 g/cm³, which corresponded to Example 3, were subjected to degreasing at 350°C for 90 minutes in the same manner as in Examples 1 to 5, and then subjected to steam treatment at the following four kinds of treatment temperatures for 40 minutes. The resultant test pieces were adopted as Examples 6 to 9.

Example 6: 350°C Example 7: 400°C Example 8: 450°C Example 9: 550°C

Examples 10 and 11

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[0032] In order to investigate the influence of a difference in treatment time of the steam treatment, two test pieces treated for different periods of time were prepared. Specifically, green compacts each having a green density of 6. 5 g/cm³, which corresponded to Example 3, were subjected to degreasing at 350°C for 90 minutes in the same manner as in Examples 1 to 5, and then subjected to steam treatment at 500°C for the following periods of time. The resultant test pieces were adopted as Examples 10 and 11.

Example 10: 20 minutes Example 11: 80 minutes

Examples 12 and 13

**[0033]** In order to investigate the influence of a difference in material, two test pieces formed of different materials were prepared. Specifically, powder was subjected to uniaxial press forming so as to have a green density of 6.5 g/cm<sup>3</sup> in the same manner as in Example 3, and then subjected to degreasing at 350°C for 90 minutes and then steam treatment at 500°C for 40 minutes in the same manner as in Examples 1 to 5. The composition of the powder used in each Example is as described below.

Example 12: powder in which 0.7 wt% of the lubricant is added to the reduced iron powder alone (Cu: 0%) Example 13: powder in which 20 wt% of the electrolytic copper powder and 0.7 wt% of the lubricant are added to the reduced iron powder

Comparative Examples 1 to 5

**[0034]** The test pieces of Example 1 to 5 in which the degreasing step at 350°C for 90 minutes and the subsequent steam treatment step were omitted, that is, the green compacts in the state directly after compacting and forming were adopted as Comparative Examples 1 to 5.

Comparative Examples 6 and 7

**[0035]** The test pieces of Example 12 and 13 in which the degreasing step at 350°C for 90 minutes and the subsequent steam treatment step were omitted, that is, the green compacts in the state directly after compacting and forming were adopted as Comparative Examples 6 and 7.

Comparative Examples 8 to 10

[0036] The three kinds of green compacts of Fe alone, Fe+2% Cu, and Fe+20% Cu described in Comparative Examples 6, 3, and 7, respectively were sintered at 1, 100°C for 30 minutes, and adopted as Comparative Examples 8 to 10, respectively.

Comparative Example 8: Fe alone (one obtained by sintering the green compact of Comparative Example 6)
Comparative Example 9: Fe+2% Cu (one obtained by sintering the green compact of Comparative Example 3)
Comparative Example 10: Fe+20% Cu (one obtained by sintering the green compact of Comparative Example 7)

#### **Evaluation Methods**

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[0037] The mechanical characteristics of the resultant test pieces were each evaluated based on the results of the measurement of radial crushing strength in conformity to JIS Z 2507. A used testing device is Autograph AG-5000A manufactured by Shimadzu Corporation. The "radial crushing strength" refers to the strength of a cylindrical sintered compact or green compact determined based on a radial crushing load by a certain method, and the "radial crushing load" refers to a load at which the cylindrical sintered compact or green compact starts to break when compressed between two planes each parallel to its axis.

[0038] Judgement criteria of the radial crushing strength are shown in Table 1. Specifically, the radial crushing strength (unit: MPa) is divided into four levels of less than 50, 50 or more and less than 100, 100 or more and less than 150, and 150 or more as shown in the left column of Table 1, and the radial crushing strengths of the four levels are represented by Symbols "×", "\(\Delta\)", "\(\O\)", and "\(\O\)" shown in the right column of Table 1, respectively.

[Table 1]

Radial crushing strength, MPa	Judgement
Less than 50	×
50 or more and less than 100	Δ
100 or more and less than 150	0
150 or more	0

[0039] In addition, the test pieces were each measured for an inner diameter and an outer diameter before and after the steam treatment through use of an image dimension measurement system (manufactured by Keyence Corporation, IM-6000), and the percentages of change amounts after the treatment with respect to the dimensions before the treatment were calculated and adopted as dimensional change rates. The higher dimensional change rate out of the dimensional change rates of the inner diameter and the outer diameter was adopted for the judgement of a dimensional change rate. [0040] Judgement criteria of the dimensional change rate are shown in Table 2. Specifically, the dimensional change rate (unit: %) is divided into three levels of  $\pm 0.1$  or more, less than  $\pm 0.1$ , and less than  $\pm 0.05$  as shown in the left column of Table 2, and the dimensional change rates of the three levels are represented by Symbols " $\Delta$ ", "o", and " $\odot$ " shown in the right column of Table 2, respectively.

[Table 2]

Dimensional change rate, %	Judgement
±0.1 or more	Δ
Less than ±0.1	0
Less than ±0.05	0

[0041] Next, the evaluation results are described.

# (a) With Regard to Green Density

**[0042]** In order to investigate the influence of the green density on the radial crushing strength and the dimensional change rate, the 2% Cu-Fe green compacts of Examples 1 to 5 having different green densities were each subjected to the steam treatment at  $500^{\circ}$ C for 40 minutes . The judgement results of the radial crushing strength and the dimensional change rate in this case are shown in Table 3.

[Table 3]

		Green		Treatment condition	Judo	gement
	Material	density, g/cm <sup>3</sup>	Treatment	(temperature , °C×retenti on time, min)	Radial crushing strength	Dimensional change rate
Example 1	Fe+2%Cu	5.3	Steam	500×40	Δ	0

(continued)

				Green		Treatment condition	Judgement	
5		Material	density, g/cm <sup>3</sup>	Treatment	(temperature , °C×retenti on time, min)	Radial crushing strength	Dimensional change rate	
	Example 2	<b>↑</b>	6.0	<b>↑</b>	<b>↑</b>	0	0	
0	Example 3	<b>↑</b>	6.5	<b>↑</b>	<b>↑</b>	0	0	
U	Example 4	<b>↑</b>	7.0	<b>↑</b>	<b>↑</b>	0	Δ	
	Example 5	<b>↑</b>	7.2	<b>↑</b>	<b>↑</b>	0	Δ	
5	Comparative Example 1	<b>↑</b>	5.3	Absent	-	×	-	
	Comparative Example 2	<b>↑</b>	6.0	<b>↑</b>	-	×	-	
0	Comparative Example 3	<b>↑</b>	6.5	<b>↑</b>	-	×	-	
	Comparative Example 4	<b>↑</b>	7.0	<b>↑</b>	-	×	-	
5	Comparative Example 5	<b>↑</b>	7.2	<b>↑</b>	-	×	-	
J	Comparative Example 9	<b>↑</b>	6.5	Sintering	1,100×30	0	Δ	

[0043] In each of Examples 1 to 5, the radial crushing strength is increased through the steam treatment. Specifically, except that the radial crushing strength of Example 1 having a green density of 5.3 g/cm³ is less than 100 MPa, the radial crushing strength of Examples 2 to 5 each having a green density of 6.0 g/cm³ or more is 100 MPa or more. However, Example 4 having a green density of 7.0 g/cm³ and Example 5 having a green density of 7.2 g/cm³ were slightly degraded in terms of the dimensional change rate. Besides, the radial crushing strength of Example 5 is lower than that of Example 4 having a green density of 7.0 g/cm³. Those results reveal that a higher green density is not always better.

[0044] In light of the test results, it is desired to set the green density to fall within a range of from  $5.0 \text{ g/cm}^3$  to  $7.6 \text{ g/cm}^3$ , preferably from  $5.3 \text{ g/cm}^3$  to  $7.2 \text{ g/cm}^3$ , more preferably  $6.0 \text{ g/cm}^3$  or more and less than  $7.0 \text{ g/cm}^3$ . In particular, the test pieces of Examples 2 and 3 each having a green density falling within a range of  $6.0 \text{ g/cm}^3$  or more and less than  $7.0 \text{ g/cm}^3$  suffice as a machine part with regard to both the radial crushing strength and the dimensional change rate. [0045] In each of Comparative Examples 1 to 5, in which the steam treatment was not performed, the radial crushing strength was less than 50 MPa. Comparative Examples 1 to 5 were not considered as measurement targets for the dimensional change rate, because neither the steam treatment nor the sintering was performed. In Comparative Example 9 having a green density of  $6.5 \text{ g/cm}^3$ , in which the sintering was performed at 1,100 °C for 30 minutes, the dimensional change rate was  $\pm 0.1\%$  or more, while the radial crushing strength was 150 MPa or more.

# (b) With Regard to Treatment Temperature

**[0046]** In order to investigate the influence of the treatment temperature of the steam treatment on the radial crushing strength and the dimensional change rate, the 2% Cu-Fe green compacts each having a green density of 6.5 g/cm<sup>3</sup> were subjected to the steam treatment at different treatment temperatures for 40 minutes. The judgement results of the radial crushing strength and the dimensional change rate in this case are shown in Table 4.

#### [Table 4]

	Material Green density, g/cm³ Treatment (temperature, °C × retention time, min)		Treatment condition	Judgement		
		Radial crushing strength	Dimensional change rate			
Example 6	Fe+2%Cu	6.5	Steam	350x40	0	0
Example 7	1	1	1	400x40	0	0
Example 8	1	1	1	450x40	0	0
Example 3	1	1	1	500x40	0	0
Example 9	1	1	1	550x40	0	0
Comparative Example 3	1	1	Absent	-	×	-
Comparative Example 9	<b>↑</b>	<b>↑</b>	Sintering	1,100×30	0	Δ

[0047] In Examples 6, 7, and 9, in which the treatment temperature was 350°C, 400°C, and 550°C, respectively, the radial crushing strength was 100 MPa or more. In Examples 8 and 3, in which the treatment temperature was 450°C and 500°C, respectively, the radial crushing strength was 150 MPa or more. The maximum dimensional change rate was less than  $\pm 0.1\%$  in each of those Examples, and was less than  $\pm 0.05\%$  in Examples 3, 8, 7, and 6, in each of which the treatment temperature was 500°C or less. In Example 6, in which the treatment temperature was 350°C, red rust (Fe<sub>2</sub>O<sub>3</sub>) was partially generated, and an Fe<sub>3</sub>O<sub>4</sub> film, which was the original target, could not be formed alone. In Examples 7, 8, 3, and 9, in each of which the treatment temperature was higher than that in Example 6, the generation of the red rust was not observed.

[0048] In light of the test results, it is desired to set the treatment temperature of the steam treatment to fall within a range of 400°C or more, preferably 400°C or more and 550°C or less, more preferably 450°C or more and 500°C or less. Comparison between the test result of Example 3, in which the treatment temperature was 500°C, and the test result of Example 9, in which the treatment temperature was 550°C, reveals that a higher treatment temperature is not always better. In particular, it was found that a treatment temperature lower than the range of from 500°C to 560°C, which was considered as the general treatment temperature range of the related-art steam treatment, was suitable with regard to both the radial crushing strength and the dimensional change rate.

# (c) With Regard to Treatment Time

**[0049]** In order to investigate the influence of the treatment time of the steam treatment on the radial crushing strength and the dimensional change rate, the 2% Cu-Fe green compacts each having a green density of 6.5 g/cm<sup>3</sup> were subjected to the steam treatment at 500°C for different treatment times. The judgement results of the radial crushing strength and the dimensional change rate in this case are shown in Table 5.

# [Table 5]

			Liar	ole oj		
	Green Treatment condition		Judg	gement		
Material		density, Treatment g/cm <sup>3</sup>		(temperature, °C ×retention time, min)	Radial crushing strength	Dimensional change rate
Example 10	Fe+2%Cu	6.5	Steam	500×20	0	0
Example 3	<b>↑</b>	<b>↑</b>	<b>↑</b>	500x40	0	0
Example 11	<b>↑</b>	<b>↑</b>	<b>↑</b>	500×80	0	0
Comparative Example 3	<b>↑</b>	<b>↑</b>	Absent	-	×	-
Comparative Example 9	<b>↑</b>	<b>↑</b>	Sintering	1,100×30	0	Δ

**[0050]** As apparent from Table 5, in each of Examples 10, 3, and 11, the radial crushing strength was 150 MPa or more and also the dimensional change rate was less than  $\pm 0.1\%$ . In light of those results, it is considered that a sufficient effect is obtained when the treatment time of the steam treatment is 20 minutes or more.

**[0051]** While the radial crushing strength was less than 50 MPa, Comparative Example 3 was not considered as a measurement target for the dimensional change rate, because neither the steam treatment nor the sintering was performed. In Comparative Example 9, the dimensional change rate was  $\pm 0.1\%$  or more, while the radial crushing strength was 150 MPa or more.

# (d) With Regard to Difference in Material

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**[0052]** In order to investigate the influence of the material on the radial crushing strength and the dimensional change rate, the green compacts each having a green density of 6.5 g/cm<sup>3</sup> were formed by using powders in which the electrolytic copper powder was added at different ratios to the reduced iron powder serving as a base material, and then subjected to the steam treatment at 500°C for 40 minutes. The judgement results of the radial crushing strength and the dimensional change rate of each test piece in this case are shown in Table 6.

[Table 6]

		Green		Treetment condition	Judgo	ement
	Material density, Treatment	Treatment condition (temperature, °C ×retention time, min)	Radial crushing strength	Dimensional change rate		
Example 12	Fe alone	6.5	Steam	500x40	0	0
Example 3	Fe+2%Cu	1	1	<b>↑</b>	0	0
Example 13	Fe+20%Cu	1	1	<b>↑</b>	0	0
Comparative Example 6	Fe alone	<b>↑</b>	Absent	-	×	-
Comparative Example 3	Fe+2%Cu	<b>↑</b>	1	-	×	-
Comparative Example 7	Fe+20%Cu	1	1	-	×	-
Comparative Example 8	Fe alone	1	Sintering	1,100x30	0	Δ
Comparative Example 9	Fe+2%Cu	1	1	<b>↑</b>	0	Δ
Comparative Example 10	Fe+20%Cu	<b>↑</b>	<b>↑</b>	<b>↑</b>	0	Δ

[0053] In Examples 12, 3, and 13, in which the ratio of the electrolytic copper powder to the reduced iron powder was 0 wt%, 2 wt%, and 20 wt%, respectively, the radial crushing strength was 100 MPa or more. It is revealed that, in those investigated compositions, a radial crushing strength of 100 MPa or more is achieved even when the ratio of iron is reduced to 80 wt%. It is also revealed that the dimensional change rate is less than  $\pm 0.1\%$  in each of the compositions. [0054] In addition, the dimensional change rate is  $\pm 0.1\%$  or more in Comparative Examples 8, 9, and 10, in each of which the sintering was performed at 1,  $100^{\circ}$ C. From this, it is revealed that the dimensional change is small when only the steam treatment is performed. While the radial crushing strength in Comparative Examples 6, 3, and 7 was less than 50 MPa, these Comparative Examples were not considered as measurement targets for the dimensional change rate, because neither the steam treatment nor the sintering was performed.

**[0055]** Next, the following test was performed in order to evaluate the friction and wear characteristics of Example of the present invention.

**[0056]** The following two kinds of test pieces were produced: the test piece of Example 2 obtained by subjecting the green compact to the steam treatment; and a test piece of Comparative Example 11 obtained by subjecting the green compact of Comparative Example 2 not subjected to the steam treatment to sintering treatment at 1,100°C for 30 minutes. The number of test pieces was set to three in each case. Those test pieces were immersed in a lubricating oil (hydraulic

action oil Shell Tellus S2 M 68, corresponding to ISO viscosity VG 68) and vacuum-impregnated therewith at 70°C for 1 hour or more

[0057] A friction and wear test was performed by using a tester illustrated in FIGS. 2. The tester includes: an arm 22 capable of rocking about a rotary shaft 21; a mating member 24 arranged below the arm 22 and fixed to a rotary shaft 23; and a felt pad 25 configured to slide with respect to the outer peripheral surface of the mating member 24. A test piece W is mounted to the lower surface of the arm 22. The mating member 24 has an outer diameter of  $\phi$  40 mm, a subsidiary curvature R of an outer diameter surface of 60 mm, a surface roughness of 0.01  $\mu$ m Ra or less, and a Vickers hardness of 780 HV or more, and is formed of, for example, hardened steel SUJ 2. The felt pad 25 is impregnated with the same lubricating oil as that with which the test piece W is impregnated. The mating member 24 was allowed to rotate at room temperature (25°C) at a hertz maximum contact surface pressure of 0.5 GPa and a rotation speed of 0.05 m/s for 30 minutes under the state in which the test piece W was pressed against the mating member 24 from above at a predetermined load by mounting a predetermined weight 26 to the arm 22. In this case, a frictional force to be generated between the test piece W and the mating member 24 was detected with a load cell 27 mounted to the arm 22. In addition, after the completion of the rotation, a specific wear amount was calculated based on the dimensions of an indentation formed on the test piece W.

**[0058]** The friction and wear characteristics of Example 2 (steam treated product) and Comparative Example 11 (sintered product) were evaluated based on the following three items obtained through the above-mentioned test.

- Convergence value of friction coefficient
- Initial running-in characteristic
- · Specific wear amount

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[0059] The "convergence value of a friction coefficient" in the test refers to an average value of friction coefficients in 10 minutes at the end of the test. In addition, the "initial running-in characteristic" refer to transition of the friction coefficient in an early stage of the test.

**[0060]** The results of the convergence value of a friction coefficient are shown in FIG. 3. As shown in FIG. 3, the convergence value of a friction coefficient of Example 2 is almost comparable to the convergence value of a friction coefficient of Comparative Example 11.

**[0061]** The results of the transition of the friction coefficient are shown in FIG. 4. As shown in FIG. 4, the transition of the friction coefficient of Example 2 differed from the transition of the friction coefficient of Comparative Example 11. Specifically, in Comparative Example 11, the friction coefficient was as high as more than 0.2 at the beginning of the test and then lowered to about 0.15 in about 1 minute, but required from about 5 minutes to about 10 minutes to converge on its final level. Meanwhile, in Example 2, the friction coefficient was low from the beginning of the test, lowered to a level comparable to that at the end of the test at 0 seconds or within several seconds, and remained at the low level. From the foregoing, it can be said that the test piece of Example 2, which is the steam treated product, has a more excellent initial running-in characteristic as compared to the test piece of Comparative Example 11, which is the sintered product.

**[0062]** The results of the specific wear amount are shown in FIG. 5. As shown in FIG. 5, the specific wear amount of Example 2 was from  $50 \times 10^{-10}$  mm<sup>3</sup>/(N·m) to  $250 \times 10^{-10}$  mm<sup>3</sup>/ (N·m), whereas the specific wear amount of Comparative Example 11 was from  $400 \times 10^{-10}$  mm<sup>3</sup>/(N·m) to  $800 \times 10^{-10}$  mm<sup>3</sup>/(N·m). As just described, the specific wear amount of Example 2 was 1/2 or less of the specific wear amount of Comparative Example 11. From the foregoing, it can be said that the test piece of Example 2, which is the steam treated product, has more excellent wear resistance as compared to the test piece of Comparative Example 11, which is the sintered product.

[0063] From the above-mentioned results, it was found that the test piece of Example 2, which was the steam treated product, had a convergence value of a friction coefficient comparable to that of the test piece of Comparative Example 11, which was the sintered product, and had a more excellent initial running-in characteristic and more excellent wear resistance as compared to the test piece of Comparative Example 11, which was the sintered product.

**[0064]** Next, a friction and wear test similar to the above-mentioned test was performed in the absence of external oil feeding by removing the felt pad 25 of FIGS. 2. The test time was set to 5 minutes. In addition, a friction coefficient at the end of the test was adopted as the "convergence value of a friction coefficient" in this test.

**[0065]** The results of the convergence value of a friction coefficient in the friction and wear test in the absence of external oil feeding are shown in FIG. 6. As shown in FIG. 6, the convergence value of a friction coefficient of Example 2 is almost comparable to the convergence value of a friction coefficient of Comparative Example 11.

**[0066]** The results of the transition of the friction coefficient in the friction and wear test in the absence of external oil feeding are shown in FIG. 7. As shown in FIG. 7, in Comparative Example 11, the friction coefficient was as high as more than 0.15 at the beginning of the test and lowered to a level comparable to the convergence value over about 1 minute. Meanwhile, in Example 2, the friction coefficient was low at almost the same level from the beginning to the end of the test. From the foregoing, it can be said that the test piece of Example 2, which is the steam treated product, has

a more excellent initial running-in characteristic as compared to the test piece of Comparative Example 11, which is the

[0067] The results of the specific wear amount in the friction and wear test in the absence of external oil feeding are shown in FIG. 8. As shown in FIG. 8, the specific wear amount of Example 2 was from 1,600×10-10 mm<sup>3</sup>/(N·m) to 2,500×10<sup>-10</sup> mm<sup>3</sup>/ (N·m), whereas the specific wear amount of Comparative Example 11 was from 3, 000x10<sup>-10</sup> mm<sup>3</sup>/ (N·m) to  $7,000\times10^{-10}$  mm<sup>3</sup>/(N·m). As just described, the specific wear amount of Example 2 was 1/2 or less of the specific wear amount of Comparative Example 11. From the foregoing, it can be said that the test piece of Example 2, which is the steam treated product, has more excellent wear resistance as compared to the test piece of Comparative Example 11, which is the sintered product.

[0068] From the above-mentioned results, it was found that, even in the case of the absence of external oil feeding, the test piece of Example 2, which was the steam treated product, had a convergence value of a friction coefficient comparable to that of the test piece of Comparative Example 11, which was the sintered product, and had a more excellent initial running-in characteristic and more excellent wear resistance as compared to the test piece of Comparative Example 11, which was the sintered product, as in the case of the presence of external oil feeding.

[0069] The embodiment of the present invention has been described above with reference to specific Examples, but the present invention is not limited to the above-mentioned embodiment. The present invention can be carried out with various modifications without departing from the scope of claims.

[0070] The machine part of the present invention has a radial crushing strength of more than 100 MPa, and hence can be used as an alternative to a related-art sintered metal part. Specific examples of the sinteredmetal part include a slide part and a magnetic core. An example of the slide part is a part configured to slide with respect to a mating member through the intermediation of a lubricating oil, and examples of such part include a bearing, a gear, and a cam to be used in an oil-lubricated environment. An example of the bearing is a plain bearing configured to slidably support a mating member (shaft) through the intermediation of an oil, and specific examples of such bearing include a sintered oil-impregnated bearing and a fluid dynamic bearing. In addition, as a matter of course, the machine part of the present invention can be used not only as an alternative to a sintered machine part for high load applications, but also as an alternative to a sintered metal part for low load applications.

## Claims

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1. A machine part, comprising a green compact obtained by press forming raw material powder containing, as a main raw material, metal powder capable of forming an oxide film, wherein:

the oxide film is formed between particles of the metal powder; and the machine part has a radial crushing strength of more than 100 MPa.

- 2. The machine part according to claim 1, wherein the green compact has a green density measured by a dimension measurement method falling within a range of from 5.0 g/cm<sup>3</sup> to 7.6 g/cm<sup>3</sup>.
- 3. The machine part according to claim 1 or 2, wherein the oxide film is formed through steam treatment.
- The machine part according to claim 3, wherein treatment temperature of the steam treatment is 400°C or more and 550°C or less.
- 5. The machine part according to any one of claims 1 to 4, wherein the machine part has a sliding surface configured to slide with respect to a mating member through intermediation of a lubricating oil.
- 6. A method of manufacturing a machine part having a radial crushing strength of more than 100 MPa, the method 50 comprising:

press forming raw material powder containing, as a main raw material, metal powder capable of forming an oxide film, to thereby provide a green compact; and forming the oxide film between particles of the metal powder forming the green compact through steam treatment.

7. The method of manufacturing a machine part according to claim 6, wherein the green compact has a green density measured by a dimension measurement method falling within a range of from 5.0 g/cm<sup>3</sup> to 7.6 g/cm<sup>3</sup>.

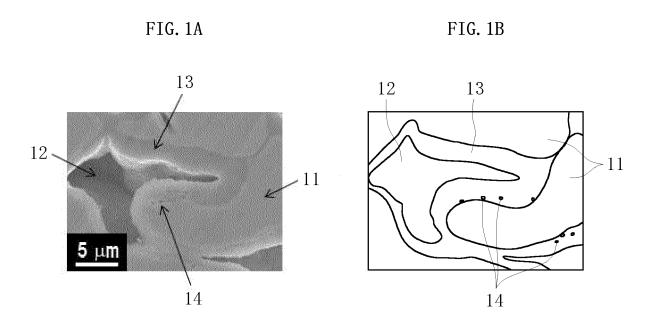
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 $\textbf{8.} \quad \text{The method of manufacturing a machine part according to claim 6 or 7, further comprising, before the steam treatment,} \\$ 

		degreasing a powder lubricant component in the green compact added for forming of the green compact.
5	9.	The method of manufacturing a machine part according to any one of claims 6 to 8, wherein treatment temperature of the steam treatment is 400°C or more and 550°C or less.
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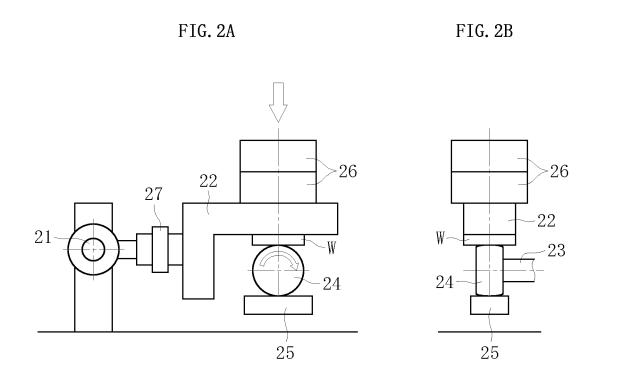


FIG. 3

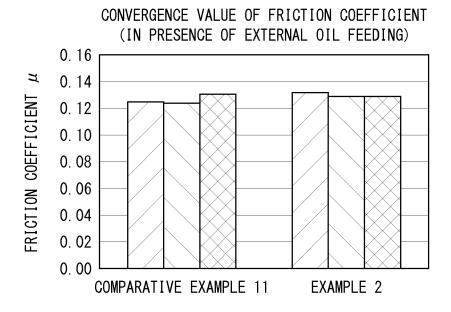


FIG. 4

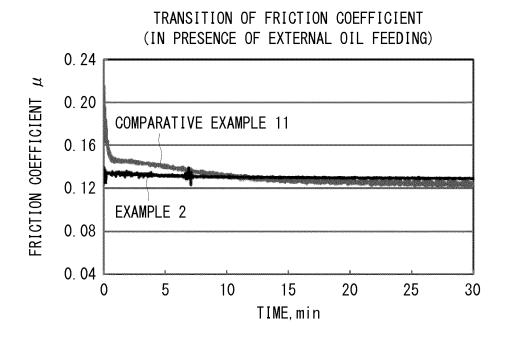


FIG. 5

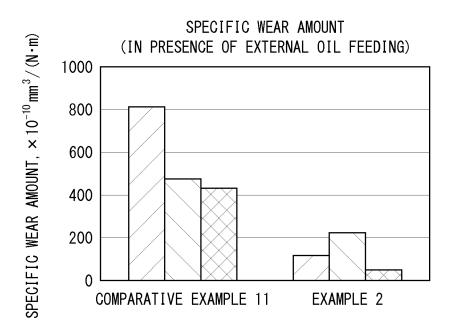


FIG. 6

CONVERGENCE VALUE OF FRICTION COEFFICIENT

# (IN ABSENCE OF EXTERNAL OIL FEEDING) 0. 16 0. 14 0. 12 0. 10 0. 08 0. 06 0. 04

EXAMPLE 2

FRICTION COEFFICIENT  $\mu$ 

0. 020. 00

COMPARATIVE EXAMPLE 11

FIG. 7

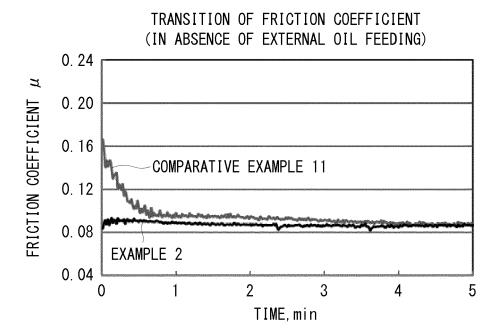
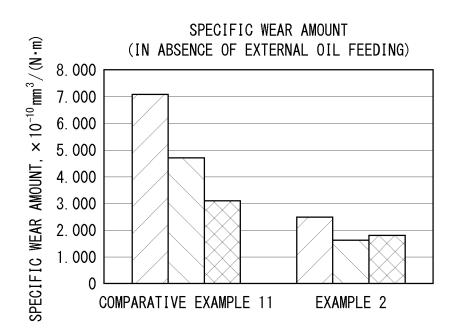


FIG. 8



#### INTERNATIONAL SEARCH REPORT International application No. PCT/JP2014/081250 A. CLASSIFICATION OF SUBJECT MATTER B22F3/24(2006.01)i, B22F5/00(2006.01)i 5 According to International Patent Classification (IPC) or to both national classification and IPC FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) 10 B22F3/24, B22F5/00 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 1996-2015 15 Kokai Jitsuyo Shinan Koho 1971-2015 Toroku Jitsuyo Shinan Koho 1994-2015 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) 20 C. DOCUMENTS CONSIDERED TO BE RELEVANT Category\* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. Χ JP 57-63602 A (Alliages Frittes SA), 1 – 9 17 April 1982 (17.04.1982), entire text 25 & EP 45706 A1 JP 2003-213308 A (Oiles Corp.), 1 - 9Α 30 July 2003 (30.07.2003), paragraphs [0008] to [0018], [0026] to [0033] (Family: none) 30 Α JP 10-73132 A (Nippon Piston Ring Co., Ltd.), 1 - 917 March 1998 (17.03.1998), paragraphs [0009] to [0011], [0015] & US 5900559 A 35 Further documents are listed in the continuation of Box C. See patent family annex. 40 Special categories of cited documents: 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 "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone 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) "L' document of particular relevance; the claimed invention cannot be 45 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 "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 document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 50 27 January 2015 (27.01.15) 03 February 2015 (03.02.15) Name and mailing address of the ISA/ Authorized officer Japan Patent Office 3-4-3, Kasumigaseki, Chiyoda-ku, 100-8915, Japan Telephone No. 55

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