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(54) Title: IRON-BASED SINTERED BODY AND METHOD OF MANUFACTURING THE SAME

(57) **Abrégé/Abstract:**

Provided is an iron-based sintered compact having excellent mechanical properties. In this sintered compact, the surface area ratio of pores does not exceed 15%, and the median diameter D50 of the pores in terms of surface area does not exceed 20µm.

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(54) Title: IRON-BASED SINTERED COMPACT AND METHOD FOR PRODUCING SAME

(54) 発明の名称: 鉄基焼結体およびその製造方法

(57) Abstract: Provided is an iron-based sintered compact having excellent mechanical properties. In this sintered compact, the surface area ratio of pores does not exceed 15%, and the median diameter D50 of the pores in terms of surface area does not exceed 20 μ m.

(57) 要約: 機械的特性に優れる鉄基焼結体を提供する。焼結体において、気孔の面積分率が 15%以下かつ気孔の面積基準のメジアン径 D50 を 20 μ m 以下とする。



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IRON-BASED SINTERED BODY AND METHOD OF MANUFACTURING THE SAME

TECHNICAL FIELD

5 **[0001]** This disclosure relates to an iron-based sintered body, and relates in particular to an iron-based sintered body suitable for manufacturing high strength sintered parts for automobiles, the sintered body having high sintered density and having reliably improved tensile strength and toughness (impact energy value) after performing the processes of carburizing, quenching, and
10 tempering on the sintered body. Further, this disclosure relates to a method of manufacturing the iron-based sintered body.

[0002] Powder metallurgical techniques enable producing parts with complicated shapes in shapes that are extremely close to product shapes (so-called near net shapes) with high dimensional accuracy, and consequently
15 significantly reducing machining costs. For this reason, powder metallurgical products are used for various machines and parts in many fields.

[0003] In recent years, there is a strong demand for powder metallurgical products to have improved toughness in terms of improving the strength for miniaturizing parts and reducing the weight thereof and safety. In particular,
20 for powder metallurgical products (iron-based sintered bodies) which are very often used for gears and the like, in addition to higher strength and higher toughness, there is also a strong demand for higher hardness in terms of wear resistance. In order to meet the above-mentioned demands, iron-based sintered bodies of which components, structures, density and the like are
25 controlled suitably are required to be developed, since the strength and toughness of an iron-based sintered body varies widely depending on those properties.

[0004] Typically, a green compact before being subjected to sintering is produced by mixing iron-based powder with alloying powders such as copper
30 powder and graphite powder and a lubricant such as stearic acid or lithium stearate to obtain mixed powder; filling a mold with the mixed powder; and compacting the powder.

 The density of a green compact obtained through a typical powder metallurgical process is usually around 6.6 Mg/m^3 to 7.1 Mg/m^3 . The green

compact is then sintered to form a sintered body which in turn is further subjected to optional sizing or cutting work, thereby obtaining a powder metallurgical product. Further, when even higher strength is required, carburizing heat treatment or bright heat treatment may be performed after
5 sintering.

[0005] Based on the components, iron-based powders used here are categorized into iron powder (e.g. iron-based powder and the like) and alloy steel powder. Further, when categorized by production method, iron-based powders are categorized into atomized iron powder and reduced iron powder.
10 Within these categories specified by production methods, the term "iron powder" is used with a broad meaning encompassing alloy steel powder as well as iron-based powder.

[0006] In terms of obtaining a sintered body with high strength and high toughness, it is advantageous that iron-based powder being a main component
15 in particular allows alloying of the powder to be promoted and high compressibility of the powder to be maintained.

First, known iron-based powders obtained by alloying include:

- (1) mixed powder obtained by adding alloying element powders to iron-based powder,
- 20 (2) pre-alloyed steel powder obtained by completely alloying alloying elements,
- (3) partially diffusion alloyed steel powder (also referred to as composite alloy steel powder) obtained by partially adding alloying element powders in a diffused manner to the surface of particles of iron-based powder or
25 pre-alloyed steel powder.

[0007] The mixed powder (1) mentioned above advantageously has high compressibility equivalent to that of pure iron powder. However, in sintering, the alloying elements are not sufficiently diffused in Fe and form a non-uniform microstructure, which would result in poor strength of the
30 resulting sintered body. Further, since Mn, Cr, V, Si, and the like are more easily oxidized than Fe, when these elements are used as the alloying elements, they get oxidized in sintering, which would reduce the strength of the resulting sintered body. In order to suppress the oxidation and reduce the amount of oxygen in the sintered body, it is necessary that the atmosphere for

sintering, and the CO₂ concentration and the dew point in the carburizing atmosphere are strictly controlled in the case of performing carburizing after sintering. Accordingly, the mixed powder (1) mentioned above cannot meet the demands for higher strength in recent years and has become unused.

5 **[0008]** On the other hand, when the pre-alloyed steel powder obtained by completely alloying the elements of (2) mentioned above is used, the alloying elements can be completely prevented from being segregated, so that the microstructure of the sintered body is made uniform, leading to stable mechanical properties. In addition, also in the case where Mn, Cr, V, Si, and
10 the like are used as the alloying elements, the amount of oxygen in the sintered body can be advantageously reduced by limiting the kind and the amount of the alloying elements. However, when the pre-alloyed steel powder is produced by atomization from molten steel, oxidation in the atomization of the molten steel and solid solution hardening of steel powder
15 due to complete alloying would be caused, which makes it difficult to increase the density of the green compact after compaction (forming by pressing). When the density of the green compact is low, the toughness of the sintered body obtained by sintering the green compact is low. Therefore, also when the pre-alloyed steel powder is used, demands for higher strength and higher
20 toughness cannot be met.

[0009] The partially diffusion alloyed steel powder (3) mentioned above is produced by adding alloying elements to iron-based powder or pre-alloyed steel powder, followed by heating under a non-oxidizing or reducing atmosphere, thereby partially diffusion bonding the alloying element powders
25 to the surface of particles of iron-based powder or pre-alloyed steel powder. Accordingly, advantages of the iron-based mixed powder of (1) above and the pre-alloyed steel powder of (2) above can be obtained.

[0010] Thus, when the partially diffusion pre-alloyed steel powder is used, oxygen in the sintered body can be reduced and the green compact can have a
30 high compressibility equivalent to the case of using pure iron powder. Therefore, the sintered body has a multi-phase structure consisting of a completely alloyed phase and a partially concentrated phase, increasing the strength of the sintered body.

[0011] As basic alloy components used in the partially diffusion alloyed steel powder, Ni and Mo are used heavily.

Ni has the effect of improving the toughness of a sintered body. Adding Ni stabilizes austenite, which allows more austenite to remain as retained austenite without transforming to martensite after quenching. Further, Ni serves to strengthen the matrix of a sintered body by solid solution strengthening.

[0012] Meanwhile, Mo has the effect of improving hardenability. Accordingly, Mo suppresses the formation of ferrite during quenching, allowing bainite or martensite to be easily formed, thereby strengthening the matrix of the sintered body. Further, Mo is contained as a solid solution in a matrix to solid solution strengthen the matrix, and forms fine carbides to strengthen the matrix by precipitation.

[0013] As an example of the mixed powder for high strength sintered parts using the above-described partially diffusion alloyed steel powder, JP 3663929 B2 (PTL 1) discloses mixed powder for high strength sintered parts obtained by mixing Ni: 1 mass% to 5 mass%, Cu: 0.5 mass% to 4 mass%, and graphite powder: 0.2 mass% to 0.9 mass% to alloy steel powder in which Ni: 0.5 mass% to 4 mass% and Mo: 0.5 mass% to 5 mass% are partially alloyed. The sintered material described in PTL 1 contains 1.5 mass% of Ni at minimum, and substantially contains 3 mass% or more of Ni according to Examples of PTL 1. This means that a large amount of Ni as much as 3 mass% or more is required to obtain a sintered body having a high strength of 800 MPa or more. Further, obtaining a material having a high strength of 1000 MPa or more by subjecting a sintered body to carburizing, quenching, and tempering also requires a large amount of Ni as much as for example 3 mass% or 4 mass%.

[0014] However, Ni is an element which is disadvantageous in terms of addressing recent environmental problems and recycling, so its use is desirably avoided as possible. Also in respect of cost, adding several mass% of Ni is significantly disadvantageous. Further, when Ni is used as an alloying element, sintering is required to be performed for a long time in order to sufficiently diffuse Ni in iron powder or steel powder. Moreover, when Ni being an austenite phase stabilizing element is not sufficiently diffused, a

high Ni concentration area is stabilized as the austenite phase (hereinafter also referred to as γ phase) and the other area where Ni is hardly contained is stabilized as other phases, resulting in a non-uniform metal structure in the sintered body.

- 5 [0015] As a Ni-free technique, JP 3651420 B2 (PTL 2) discloses a technique associated with partially diffusion alloyed steel powder of Mo free of Ni. That is, PTL 2 states that optimization of the Mo content results in a sintered body having high ductility and high toughness that can resist repressing after sintering.
- 10 [0016] Further, regarding a high density sintered body free of Ni, JP H04-285141 A (PTL 3) discloses mixing iron-based powder having a mean particle diameter of 1 μm to 18 μm with copper powder having a mean particle diameter of 1 μm to 18 μm at a weight ratio of 100:(0.2 to 5), and compacting the mixed powder and sintering the green compact. In the technique
- 15 disclosed in PTL 3, iron-based powder having a mean particle diameter that is extremely smaller than that of typical one is used, so that a sintered body having a density as extremely high as 7.42 g/cm^3 or more can be obtained.
- [0017] WO 2015/045273 A1 (PTL 4) discloses that a sintered body having high strength and high toughness is obtained using powder free of Ni, in
- 20 which Mo is adhered to the surface of iron-based powder particles by diffusion bonding to achieve a specific surface area of 0.1 m^2/g or more.
- [0018] Further, JP 2015-014048 A (PTL 5) discloses that a sintered body having high strength and high toughness is obtained using powder in which Mo is adhered to iron-based powder particles containing reduced iron powder
- 25 by diffusion bonding.
- [0019] JP 2015-004098 A (PTL 6) describes that Fe-Mn-Si powder is added to iron powder particles of a small particle size and the mixed powder is warm compacted in a lubricated mold, thereby reducing the maximum pore length of the sintered body to obtain a sintered body having high strength and high
- 30 toughness.

CITATION LIST

Patent Literature

[0020] PTL 1: JP 3663929 B2

PTL 2: JP 3651420 B2

PTL 3: JP H04-285141 A

PTL 4: WO 2015/045273 A1

PTL 5: JP 2015-014048 A

5 PTL 6: JP 2015-004098 A

SUMMARY

(Technical Problem)

10 [0021] However, the sintered materials obtained in accordance with the description of PTL 2, PTL 3, PTL 4, PTL 5, and PTL 6 above have been found to have the following respective problems.

[0022] The technique disclosed in PTL 2 is designed to achieve high strength by recompression after sintering. Accordingly, when a sintered material is manufactured by a typical metallurgical process, both sufficient strength and
15 toughness are hardly achieved at the same time.

[0023] Further, the iron-based powder used for the sintered material described in PTL 3 has a mean particle diameter of 1 μm to 18 μm which is smaller than normal. Such a small particle diameter results in poor flowability of the mixed powder inducing cracking and chipping of the green compact due to unevenness of the powder in filling the mold. Therefore, it is
20 difficult to obtain a sintered body having sufficient strength and toughness.

[0024] Further, since the powder described in PTL 4 has extremely large specific surface area, use of such powder results in low flowability of the powder and induces cracking and chipping of the green compact due to unevenness of the powder in filling the mold. Therefore, it is difficult to
25 obtain a sintered body having sufficient strength and toughness.

[0025] Also for the sintered body described in PTL 5, as with the technique described in PTL 4, reduced iron powder having extremely large specific surface area is used, which results in low flowability of the powder and
30 induces cracking and chipping of the green compact due to unevenness of the powder in filling the mold. Therefore, it is difficult to obtain a sintered body having sufficient strength and toughness.

[0026] The toughness of the sintered body disclosed in PTL 6 is increased mainly by limiting the maximum pore length; however, high strength and

toughness are hardly achieved by only limiting the maximum pore length, and further improvement is required.

[0027] It could be helpful to provide an iron-based sintered body having excellent mechanical properties as well as a method of manufacturing the same.

(Solution to Problem)

[0028] With a view to achieve the above objective, we made various studies to obtain a sintered body having both high strength and high toughness. As a result, we discovered the following:

10 for an iron-based sintered body obtained by pressing mixed powder made of iron-based powder and additives and then sintering, adjusting the mean diameter of pores in the sintered body contributes to the improvement in the impact energy value due to the dispersion of stress concentrations in the structure.

15 [0029] This disclosure is based on the aforementioned discoveries and further studies. Specifically, the primary features of this disclosure are described below.

1. An iron-based sintered body, comprising an area fraction of pores in the iron-based sintered body of 15 % or less, and an area-based median size D50 of the pores of 20 μm or less.

[0030] 2. The iron-based sintered body according to 1. above, comprising Mo, Cu, and C.

[0031] 3. The iron-based sintered body according to 2. above, comprising Mo in an amount of 0.2 mass% to 1.5 mass%, Cu in an amount of 0.5 mass% to 4.0 mass%, and C in an amount of 0.1 mass% to 1.0 mass%.

[0032] 4. The iron-based sintered body according to any one of 1. to 3. above, wherein the iron-based sintered body has been carburized, quenched, and tempered.

[0033] 5. A method of manufacturing an iron-based sintered body, the method comprising: compacting (i) partially diffusion alloyed steel powder in which Mo is adhered to the surface of particles of iron-based powder by diffusion bonding with (ii) mixed powder for powder metallurgy obtained by mixing at least Cu powder and graphite powder at a pressure of 400 MPa or more to

obtain a compact; and then sintering the obtained compact at 1000 °C or higher for 10 min or more.

[0034] 6. The method of manufacturing a high strength according to 5. above, the method further comprising carburizing, quenching, and tempering after
5 sintering the obtained compact.

[0035] 7. The method of manufacturing an iron-based sintered body, according to 5. or 6. above, wherein the mixed powder for powder metallurgy contains Mo in an amount of 0.2 mass% to 1.5 mass% and the balance consisting of Fe and incidental impurities.

10 [0036] 8. The method of manufacturing an iron-based sintered body, according to any one of 5. to 7. above, wherein the partially diffusion alloyed steel powder has a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m^2/g , and a circularity of particles of the partially diffusion alloyed steel powder that have a diameter in a range of 50 μm to 100 μm is 0.65
15 or less.

[0037] 9. The method of manufacturing an iron-based sintered body, according to any one of 5. to 8. above, wherein the amount of the Cu powder mixed is 0.5 mass% to 4.0 mass% of the mixed powder for powder metallurgy.

(Advantageous Effect)

20 [0038] This disclosure can provide an iron-based sintered body having both high strength and high toughness.

[0038a] Also disclosed is an iron-based sintered body, comprising:

partially diffusion alloyed steel powder in which Mo is adhered to the surface of particles of iron-based powder;

25 Cu powder; and
graphite powder; wherein

the partially diffusion alloyed steel powder has a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m^2/g , and a circularity of particles of the partially diffusion alloyed steel powder that have a
30 diameter in a range of 50 μm to 100 μm is 0.65 or less,

the iron-based sintered body has a chemical composition consisting of:

Mo in an amount of 0.2 mass% to 1.5 mass%,

Cu in an amount of 0.5 mass% to 4.0 mass%,

C in an amount of 0.1 mass% to 1.0 mass%, and

5 the balance consisting of Fe and incidental impurities, and

the iron-based sintered body has an area fraction of pores in the iron-based sintered body more than 0% and of 15 % or less, and an area-based median size D50 of the pores of 20 μm or less.

[0038b] Also disclosed is a RV3 A method of manufacturing an iron-based
10 sintered body, the method comprising:

(a) a step of diffusion bonding of iron-based powder with Mo to obtain partially diffusion alloyed steel powder in which Mo is adhered to the surface of particles of iron-based powder;

(b) a step of mixing at least Cu powder and graphite powder with the
15 partially diffusion alloyed steel powder—to obtain mixed powder for powder metallurgy;

(c) a step of compacting the mixed powder for powder metallurgy at a pressure of 400 MPa or more to obtain a compact; and

(d) then sintering the obtained compact at 1000 °C or higher for 10 min
20 or more;
wherein

the partially diffusion alloyed steel powder has a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m^2/g , and a circularity of particles of the partially diffusion alloyed steel powder that have a
25 diameter in a range of 50 μm to 100 μm is 0.65 or less, and

the obtained iron-based sintered body has a chemical composition consisting of:

Mo in an amount of 0.2 mass% to 1.5 mass%,

Cu in an amount of 0.5 mass% to 4.0 mass%,

30 C in an amount of 0.1 mass% to 1.0 mass%, and

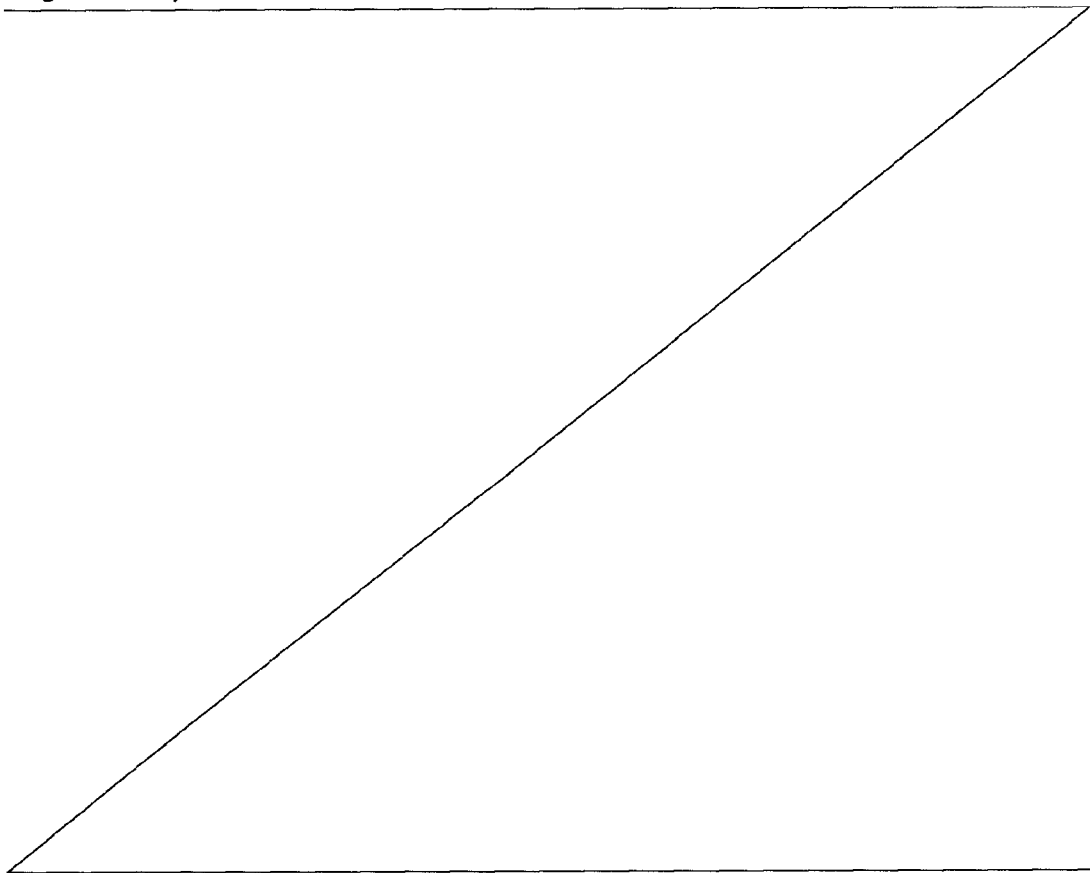
the balance consisting of Fe and incidental impurities.

DETAILED DESCRIPTION

[0039] Our methods and products will be described in detail below.

The area fraction of pores in the disclosed sintered body is 15 % or less and the area-based median size D50 of the pores is 20 μm or less.

- 5 [0040] Pores are unavoidably formed in the iron-sintered body obtained by sintering a green compact obtained by compacting alloy steel powder for powder metallurgy, and it is important to control the pores for improving the strength and toughness of the sintered body. That is, since smaller pores hardly act as starting points of cracks, it is important that the area-based median size D50 of
- 10 the pores is 20 μm or less. More preferably, the area-based median size D50 is 15 μm or less. When the median size D50 exceeds 20 μm , the toughness is significantly reduced.



[0041] Here, the median size D50 of the pores can be measured in the following manner.

First, a sintered body is embedded in a thermosetting resin. A cross section is then mirror-polished and the cross section is imaged using an optical microscope at 100× magnification over a field of view of 843 μm × 629 μm. The cross-sectional area A of all the pores in 20 fields randomly selected from the resulting micrograph of the cross section is measured. The equivalent circle diameter d_c that is the diameter of a circle having an area equal to the measured cross-sectional area is determined in accordance with the following equation (I). Next, the areas of the pores are integrated in ascending order of the circle equivalent diameter and a circle equivalent diameter at which the integrated value is 50 % of the total area of the pores is defined as an area-based median size D50.

$$d_c = 2\sqrt{A/\pi} \quad \cdots (I)$$

[0042] As described above, the median size D50 of the pores of the sintered body is controlled to 20 μm or less, since a median size D50 exceeding 20 μm increases pores having an indefinite shape and such pores become stress concentrations when deformation occurs, which reduces strength and toughness.

Here, in order to control the area fraction of the pores in the sintered body to 15 % or less and the median size D50 of the pores to 20 μm or less, partially diffusion alloyed steel powder or mixed powder for powder metallurgy which is a material of the sintered body is used. The partially diffusion alloyed steel powder is obtained by adhering Mo powder particles to the surface of iron-based powder particles, the steel powder particles having a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m²/g, and particles of the steel powder that have a diameter in a range of 50 μm to 100 μm has a circularity of 0.65. Thus, sintering is promoted in manufacturing a sintered body to be described, so that a desired sintered body can be obtained.

[0043] Since the number of pores is preferably smaller, the area fraction of the pores in the sintered body is controlled to 15 % or less. This is because since an area fraction of the pores exceeding 15 % reduces the content of metal in the sintered body, even if the pore diameter is reduced, sufficient

strength and toughness cannot be obtained. Note that making the pores in the sintered body be 0 % requires significant effort and is not realistic. The pores in the sintered body obtained by the following method is at least approximately 5 %.

- 5 [0044] Here, the area fraction of the pores in the sintered body can be calculated by the following method.

In a manner similar to the above, the cross-sectional area A of all the pores in 20 fields is measured and summed to find the total pore area A_t of all the observed fields. Dividing A_t by the total of the areas of all the observed
10 fields gives the area fraction of the pores.

[0045] Further, the length of the pores in the sintered body is preferably smaller. The "mean maximum pore length" that is an indicator of the length of the pores is calculated as follows. First, the maximum value of the distance between two points on the circumferential edge of each pore in the
15 field of the above micrograph of the cross section is found by image analysis and is defined as the "pore length" of the pore. The "maximum pore length" is longest among the "pore lengths" of all the pores included in a field of view of the micrograph of the cross section. Further, the "mean maximum pore length" is the arithmetic mean value of the maximum pore lengths found for
20 20 fields selected randomly. Note that in order to achieve sufficient mechanical properties, the mean maximum pore length is preferably less than 100 μm .

[0046] Further, the above sintered body preferably contains Mo, Cu, and C. Mo has the effect of improving hardenability. Cu has the effect of improving
25 solid solution strengthening and hardenability of iron-based powder. C has the effect of enhancing the strength of iron-based sintered body by being precipitated as a solid solution or fine carbide in iron. Preferred content range of the respective elements contained in the disclosed iron-based sintered body is Mo: 0.2 mass% to 1.5 mass%, Cu: 0.5 mass% to 4.0 mass%, and C: 0.1
30 mass% to 1.0 mass%. When the elements are less than the above range, the strength cannot sufficiently be increased, whereas when they are added to be more than the above range, the structure is extremely hardened and the toughness is reduced.

[0047] Next, a method of obtaining the above sintered body will be described. The following method is a mere example, and the disclosed iron-based sintered body may be obtained by a method other than the following method.

5 In manufacturing a sintered body by sintering a green compact obtained by compacting mixed powder for powder metallurgy, the mixed powder is made into the green compact by compaction using a punch by a technique in which the compaction is performed while rotating the punch about an imaginary axis in the pressing direction. This method can produce more shear strains in the mixed powder than in typical compaction, 10 facilitating plastic deformation of the mixed powder, and the pores in the sintered body can have a finer diameter.

[0048] Next, a method of manufacturing a sintered body, particularly suitable for manufacturing a sintered body containing Mo, Cu, and C will be described.

15 In this method, mixed powder for powder metallurgy containing iron-based powder and additives is compacted by a conventional method to form a green compact, and the green compact is then sintered by a conventional method, thereby obtaining an iron-based sintered body. On this occasion, with a view to increasing the density of the sintered body, it is preferable that Mo-concentrated portions are formed in sintered neck parts 20 between particles of the iron-based powder in the green compact; iron-based powder having particles with low circularity is used to achieve stronger entanglement between particles of the powder during compaction thereby promoting sintering; and the sintering is also promoted with suppressed Cu growth. When the density of a sintered body is high, both strength and 25 toughness are improved; however, since a sintered body obtained by this manufacturing method has a uniform metal structure, the mechanical properties of the sintered body are stable with little variation, unlike conventional sintered bodies, for example, those using Ni.

30 [0049] In order to obtain such a sintered body, the sintered body is preferably manufactured using partially diffusion alloyed steel powder described below as the iron-based powder of the above mixed powder for powder metallurgy.

Mixed powder for powder metallurgy preferably used in this disclosure is obtained by mixing partially diffusion alloyed steel powder in

which Mo is adhered by diffusion bonding to the surface of particles of iron-based powder of which mean particle diameter, circularity, and specific surface area are appropriate (hereinafter also referred to as partially alloyed steel powder) with an appropriate amount of Cu powder having a mean
5 particle diameter in a range described below as well as graphite powder.

[0050] Mixed powder for powder metallurgy according to this disclosure will now be described in detail. Note that "%" herein means "mass%" unless otherwise specified. Accordingly, the Mo content, the Cu content, and the graphite powder content each represents the proportion of the element in the
10 entire mixed powder for powder metallurgy (100 mass%).

[0051] (Iron-based powder)

As described above, the partially diffusion alloyed steel powder is obtained by adhering Mo to the surface of particles of the iron-based powder, and it is preferred that the mean particle diameter is 30 μm to 120 μm , the
15 specific surface area is less than 0.10 m^2/g , and particles having a diameter in a range of 50 μm to 100 μm have a circularity (cross-sectional circularity) of 0.65 or less. Here, when the iron-based powder is partially alloyed, the particle diameter and the circularity hardly change. Accordingly, iron-based powder having a mean particle diameter and a circularity in the same range as
20 that of the partially diffusion alloyed steel powder is used.

[0052] First, the iron-based powder preferably has a mean particle diameter of 30 μm to 120 μm and particles having a diameter in a range of 50 μm to 100 μm preferably have a circularity (roundness of the cross section) of 0.65 or less. For the reasons described below, the partially alloyed steel powder
25 is required to have a mean particle diameter of 30 μm to 120 μm and particles having a diameter in a range of 50 μm to 100 μm are required to have a circularity of 0.65 or less. Accordingly, the iron-based powder is also required to meet those conditions.

[0053] Here, the mean particle diameter of the iron-based powder and the
30 partially alloyed steel powder refers to the median size D50 determined from the cumulative weight distribution, and is a particle diameter found by determining the particle size distribution using a sieve according to JIS Z 8801-1, producing the integrated particle size distribution from the resulting particle size distribution, and finding the particle diameter obtained when the

oversized particles and the undersized particles constitute 50 % by weight each.

[0054] Further, the circularity of the particles of iron-based powder and partially alloyed steel powder can be determined as follows. Although a case of iron-based powder is explained by way of example, the circularity of partially alloyed steel powder particles is also determined through the same process.

First, iron-based powder is embedded in a thermosetting resin. On this occasion, the iron-based powder is embedded to be uniformly distributed in an area with a thickness of 0.5 mm or more in the thermosetting resin so that a sufficient number of cross sections of the iron-based powder particles can be observed in an observation surface exposed by polishing the powder-embedded resin. After that, the resin is polished to expose a cross section of the iron-based powder particles; the cross section of the resin is mirror polished; and the cross section is magnified and imaged by an optical microscope. The cross sectional area A and the peripheral length Lp of the iron-based powder particles in the resulting micrograph of the cross section are determined by image analysis. Examples of software capable of such image analysis include ImageJ (open source, National Institutes of Health). The circle equivalent diameter dc is calculated from the determined cross-sectional area A. Here, dc is calculated by the same equation (I) as in the case of the pores.

$$d_c = 2\sqrt{A/\pi} \quad \cdot \cdot \cdot (I)$$

[0055] Next, the peripheral length of a circular approximation of each powder particle Lc is calculated by multiplying the particle diameter dc by the number π . The circularity C is calculated from the determined Lc and the peripheral length Lp of the cross section of each iron-based powder particle. Here, the circularity C is a value defined by the following equation (II).

When the circularity C is 1, the cross-sectional shape of the particle is a perfect circle, and a smaller C value results in a more indefinite shape.

$$C = L_c/L_p \quad \cdot \cdot \cdot (II)$$

[0056] Note that iron-based powder means powder having an Fe content of 50 % or more. Examples of iron-based powder include as-atomized powder (atomized iron powder as atomized), atomized iron powder (obtained by

reducing as-atomized powder in a reducing atmosphere), and reduced iron powder. In particular, iron-based powder used in this disclosure is preferably as-atomized powder or atomized iron powder. This is because since reduced iron powder contains many pores in the particles, sufficient
5 density would not be obtained during compaction. Further, reduced iron powder contains more inclusions acting as starting points of fracture in the particles than atomized iron powder, which would reduce the fatigue strength which is one of the important mechanical properties of a sintered body.

[0057] Specifically, iron-based powder preferably used in this disclosure is
10 any one of as-atomized powder obtained by atomizing molten steel, drying the atomized molten steel, and classifying the resulting powder without performing heat treatment for e.g., deoxidation (reduction) and decarbonization; and atomized iron powder obtained by reducing as-atomized powder in a reducing atmosphere.

15 Iron-based powder satisfying the above-described circularity can be obtained by appropriately adjusting the spraying conditions for atomization and conditions for additional processes performed after the spraying. Further, iron-based powder having particles of different circularities may be mixed and the circularity of the particles of the iron-based powder that have a
20 particle diameter in a range of 50 μm to 100 μm may be controlled to fall within the above-described range.

[0058] (Partially diffusion alloyed steel powder)

Partially diffusion alloyed steel powder is obtained by adhering Mo to the surface of particles of the above iron-based powder, and it is required that
25 the mean particle diameter is 30 μm to 120 μm , the specific surface area is less than 0.10 m^2/g , and particles having a diameter in a range of 50 μm to 100 μm have a circularity of 0.65 or less.

[0059] Thus, the partially diffusion alloyed steel powder is produced by adhering Mo to the above iron-based powder by diffusion bonding. The Mo
30 content is set to be 0.2 % to 1.5 % of the entire mixed powder for powder metallurgy (100 %). When the Mo content is less than 0.2 %, the hardenability and strength of a sintered body manufactured using the mixed powder for powder metallurgy are poorly improved. On the other hand, when the Mo content exceeds 1.5 %, the effect of improving hardenability

reaches a plateau, and the structure of the sintered body becomes rather non-uniform. Accordingly, high strength and toughness cannot be obtained. Therefore, the content of Mo adhered by diffusion bonding is set to be 0.2 % to 1.5 %. The Mo content is preferably 0.3 % to 1.0 %, more preferably 0.4 % to 0.8 %.

[0060] Here, Mo-containing powder can be given as an example of a Mo source. Examples of the Mo-containing powder include pure metal powder of Mo, oxidized Mo powder, and Mo alloy powders such as Fe-Mo (ferromolybdenum) powder. Further, Mo compounds such as Mo carbides, Mo sulfides, and Mo nitrides can be used as preferred Mo-containing powders. These material powders can be used alone; alternatively, some of these material powders can be used in a mixed form.

[0061] Specifically, the above-described iron-based powder and the Mo-containing powder are mixed in the proportions described above (the Mo content is 0.2 % to 1.5 % of the entire mixed powder for powder metallurgy (100 %)). The mixing method is not particularly limited, and the powders can be mixed by a conventional method using a Henschel mixer, a cone blender, or the like.

[0062] Next, mixed powder of the above-described iron-based powder and the Mo-containing powder is heated so that Mo is diffused in the iron-based powder through the contact surface between the iron-based powder and the Mo-containing powder, thereby joining Mo to the iron-based powder. Partially alloyed steel powder containing Mo can be obtained by this heat treatment.

As the atmosphere for diffusion-bonding heat treatment, a reducing atmosphere or a hydrogen-containing atmosphere is preferable, and a hydrogen-containing atmosphere is particularly suitable. Alternatively, the heat treatment may be performed under vacuum.

Further, for example when a Mo compound such as oxidized Mo powder is used as the Mo-containing powder, the temperature of the heat treatment is preferably set to be in a range of 800 °C to 1100 °C. When the temperature of the heat treatment is lower than 800 °C, the Mo compound is insufficiently decomposed and Mo is not diffused into the iron-based powder, so that Mo hardly adheres to the iron-based powder. When the heat

treatment temperature exceeds 1100 °C, sintering between iron-based powder particles is promoted during the heat treatment, and the circularity of the iron-based powder particles exceeds the predetermined range. On the other hand, when a metal and an alloy, for example, Mo pure metal and an alloy
5 such as Fe-Mo are used for the Mo-containing powder, a preferred heat treatment temperature is in a range of 600 °C to 1100 °C. When the temperature of the heat treatment is lower than 600 °C, Mo is not sufficiently diffused into the iron-based powder, so that Mo hardly adheres to the iron-based powder. On the other hand, when the heat treatment temperature
10 exceeds 1100 °C, sintering between iron-based powder particles is promoted during the heat treatment, and the circularity of the partially alloyed steel powder exceeds the predetermined range.

[0063] When heat treatment, that is, diffusion bonding is performed as described above, since partially alloyed steel powder particles are usually
15 sintered together and solidified, grinding and classification are performed to obtain particles having a predetermined particle diameter described below. Specifically, in order to achieve the predetermined particle diameter, the grinding conditions are tightened or coarse powder is removed by classification using a sieve with openings of a predetermined size, as
20 necessary. In addition, annealing may optionally be performed.

[0064] Specifically, it is important that the mean particle diameter of the partially alloyed steel powder is in a range of 30 µm to 120 µm. The lower limit of the mean particle diameter is preferably 40 µm, more preferably 50 µm. Meanwhile, the upper limit of the mean particle diameter is preferably
25 100 µm, more preferably 80 µm.

As described above, the mean particle diameter of the partially alloyed steel powder refers to the median size D50 determined from the cumulative weight distribution, and is a particle diameter found by determining the particle size distribution using a sieve according to JIS Z 8801-1, producing
30 the integrated particle size distribution from the resulting particle size distribution, and finding the particle diameter obtained when the oversized particles and the undersized particles constitute 50 % by weight each.

Here when the mean particle diameter of the partially alloyed steel powder particles is smaller than 30 µm, the flowability of the partially alloyed

steel powder is reduced, and for example the productivity in compaction using a mold is affected. On the other hand, when the mean particle diameter of the partially alloyed steel powder particles exceeds 120 μm , the driving force is weakened during sintering and coarse pores are formed around the coarse iron-based powder particles. This reduces the sintered density and leads to reduction in the strength and toughness of a sintered body and the sintered body having been carburized, quenched, and tempered. The maximum particle diameter of the partially alloyed steel powder particles is preferably 180 μm or less.

10 [0065] Further, in terms of compressibility, the specific surface area of the partially alloyed steel powder particles is set to be less than $0.10 \text{ m}^2/\text{g}$. Here, the specific surface area of the partially alloyed steel powder refers to the specific surface area of particles of the partially alloyed steel powder except for additives (Cu powder, graphite powder, lubricant).

15 [0066] When the specific surface area of the partially alloyed steel powder exceeds $0.10 \text{ m}^2/\text{g}$, the flowability of the mixed powder for powder metallurgy is reduced. Note that the lower limit of the specific surface area is not specified; however, the lower limit of the specific surface area achieved industrially is approximately $0.010 \text{ m}^2/\text{g}$. The specific surface area can be controlled as desired by adjusting the particle size of coarse particles of more than 100 μm and fine particles of less than 50 μm after diffusion bonding by sieving. Specifically, the specific surface area is reduced by reducing the proportion of fine particles or increasing the proportion of coarse particles.

25 [0067] Further, particles of the partially alloyed steel powder that have a diameter of 50 μm to 100 μm are required to have a circularity of 0.65. The circularity is preferably 0.60 or less, more preferably 0.58 or less. Reducing the circularity increases the entanglement between particles during compaction and improves the compressibility of the mixed powder for powder metallurgy, so that coarse pores in the green compact and the sintered body are reduced. On the other hand, an excessively low circularity reduces the compressibility of the mixed powder for powder metallurgy. Accordingly, the circularity is preferably 0.40 or more.

30 [0068] The circularity of the partially alloyed steel powder particles having a diameter of 50 μm to 100 μm can be measured as follows. First, the particle

diameter of the partially alloyed steel powder particles is calculated in the same manner as that of the above-described iron-based powder particles and is expressed as d_c , and the partially alloyed steel powder particles having d_c in a range of 50 μm to 100 μm are extracted. Here, optical microscopy imaging performed is such that at least 150 particles of the partially alloyed steel powder that have a diameter in a range of 50 μm to 100 μm can be extracted. The circularity of the extracted partially alloyed steel powder particles was calculated in the same manner as in the case of the above-described iron-based powder.

Note that the particle diameter of the partially alloyed steel powder particles is limited to 50 μm to 100 μm because reducing the circularity of the particles of this range can most effectively promote sintering. Specifically, since particles of less than 50 μm are fine particles which originally facilitate sintering, reducing the circularity of such particles of less than 50 μm does not significantly promote sintering. Further, since particles having a particle diameter exceeding 100 μm are extremely coarse, reducing the circularity of those particles does not significantly promote sintering.

[0069] In this disclosure, the remainder components in the partially alloyed steel powder are iron and inevitable impurities. Here, impurities contained in the partially alloyed steel powder may be C (except for graphite content), O, N, S, and others, the contents of which may be set to C: 0.02 % or less, O: 0.3 % or less, N: 0.004 % or less, S: 0.03 % or less, Si: 0.2 % or less, Mn: 0.5 % or less, and P: 0.1 % or less in the partially alloyed steel powder without any particular problem. The content of O, however, is preferably 0.25 % or less. It should be noted that when the amount of incidental impurities exceeds the above range, the compressibility in compaction using the partially alloyed steel powder decreases, which makes it difficult to obtain a green compact having sufficient density by the compaction.

[0070] In this disclosure, a sintered body manufactured using mixed powder for powder metallurgy is further subjected to carburizing, quenching, and tempering, and Cu powder and graphite powder are then added to the partially alloyed steel powder obtained as described above for the purpose of achieving a tensile strength of 1000 MPa.

[0071] (Cu powder)

Cu is an element useful in improving the solid solution strengthening and the hardenability of iron-based powder thereby increasing the strength of sintered parts. The amount of Cu added is preferably 0.5 % or more and 4.0 or less. When the amount of Cu powder added is less than 0.5 %, the advantageous effects of adding Cu are hardly obtained. On the other hand, when the Cu content exceeds 4.0 %, not only does the effects improving the strength of the sintered parts reach a plateau but also the density of the sintered body is reduced. Therefore, the amount of Cu powder added is limited to a range of 0.5 % to 4.0 %. The amount added is preferably in a range of 1.0 % to 3.0 %.

[0072] Further, when Cu powder of large particle size is used, in sintering a green compact of mixed powder for powder metallurgy, molten Cu penetrates between particles of the partially alloyed steel powder to expand the volume of the sintered body after sintering, which would reduce the density of the sintered body. In order to prevent the density of the sintered body from decreasing in such a way, the mean particle diameter of the Cu powder is preferably set to be 50 μm or less. More preferably, the mean particle diameter of the Cu powder is 40 μm or less, still more preferably 30 μm or less. Although the lower limit of the mean particle diameter of the Cu powder is not specified, the lower limit is preferably set to be approximately 0.5 μm in order not to increase the production cost of the Cu powder unnecessarily.

[0073] The mean particle diameter of the Cu powder can be calculated by the following method.

Since the mean particle diameter of particles having a mean particle diameter of 45 μm or less is difficult to be measured by means of sieving, the particle diameter is measured using a laser diffraction/scattering particle size distribution measurement system. Examples of the laser diffraction/scattering particle size distribution measurement system include LA-950V2 manufactured by HORIBA, Ltd. Of course, other laser diffraction/scattering particle size distribution measurement systems may be used; however, for performing accurate measurement, the lower limit and the upper limit of the measurable particle diameter range of the system used are preferably 0.1 μm or less and 45 μm or more, respectively. Using the system

mentioned above, a solvent in which Cu powder is dispersed is exposed to a laser beam, and the particle size distribution and the mean particle diameter of the Cu powder are measured from the diffraction and scattering intensity of the laser beam. For the solvent in which the Cu powder is dispersed, ethanol is preferably used, since particles are easily dispersed in ethanol, and ethanol is easy to handle. When a solvent in which the Van der Waals force is strong and particles are hardly dispersed, such as water is used, particles agglomerate during the measurement, and the measurement result includes a mean particle diameter larger than the real mean particle diameter. Therefore, such a solvent is not preferred. Accordingly, it is preferable that Cu powder introduced into an ethanol solution is preferably dispersed using ultrasound before the measurement.

Since the appropriate dispersion time varies depending on the target powder, the dispersion is performed in 7 stages at 10 min intervals between 0 min and 60 min, and the mean particle diameter of the Cu powder is measured after each dispersion time stage. In order to prevent particle agglomeration, during each measurement, the measurement is performed with the solvent being stirred. Of the particle diameters obtained through the seven measurements performed by changing the dispersion time by 10 min, the smallest value is used as the mean particle diameter of the Cu powder.

[0074] (Graphite powder)

Graphite powder is useful in increasing strength and fatigue strength, and graphite powder is added to the partially alloyed steel powder in an amount in a range of 0.1 % to 1.0 %, and mixing is performed. When the amount of graphite powder added is less than 0.1 %, the above advantageous effects cannot be obtained. On the other hand, when the amount of graphite powder added exceeds 1.0 %, the sintered body becomes hypereutectoid, and cementite is precipitated, resulting in reduced strength. Therefore, the amount of graphite powder added is limited to a range of 0.1 % to 1.0 %. The amount of graphite powder added is preferably in a range of 0.2 % to 0.8 %. Note that the particle diameter of graphite powder to be added is preferably in a range of approximately from 1 μm to 50 μm .

[0075] In this disclosure, the Cu powder and graphite powder described above are mixed with partially diffusion alloyed steel powder to which Mo is

diffusionally adhered to obtain Fe-Mo-Cu-C-based mixed powder for powder metallurgy, and the mixing may be performed in accordance with conventional powder mixing methods.

5 [0076] Further, in a stage where a sintered body is obtained, if the sintered body needs to be further formed into the shape of parts by cutting work or the like, powder for improving machinability, such as MnS is added to the mixed powder for powder metallurgy in accordance with conventional methods.

[0077] Next, the compacting conditions and sintering conditions preferable for manufacturing a sintered body using the-above described mixed powder
10 for powder metallurgy will be described.

In compaction using the above mixed powder for powder metallurgy, a lubricant powder may also be mixed in. Further, compaction may be performed with a lubricant being applied or adhered to a mold. In either case, as the lubricant, any of metal soap such as zinc stearate and lithium stearate,
15 amide-based wax such as ethylenebisstearamide, and other well known lubricants may suitably be used. When mixing the lubricant, the amount thereof is preferably around from 0.1 parts by mass to 1.2 parts by mass with respect to 100 parts by mass of the mixed powder for powder metallurgy.

[0078] In manufacturing a green compact by compacting the disclosed mixed
20 powder for powder metallurgy, the compaction is preferably performed at a pressure of 400 MPa to 1000 MPa. When the compacting pressure is less than 400 MPa, the density of the resulting green compact is reduced, and the properties of the sintered body are degraded. On the other hand, a compacting pressure exceeding 1000 MPa extremely shortens the life of the
25 mold, which is economically disadvantageous. The compacting temperature is preferably in a range of room temperature (approximately 20 °C) to approximately 160 °C.

[0079] Further, the green compact is sintered preferably at a temperature in a range of 1100 °C to 1300 °C. When the sintering temperature is lower than
30 1100 °C, sintering stops; accordingly, it is difficult to achieve the desired tensile strength: 1000 MPa or more. On the other hand, a sintering temperature higher than 1300 °C extremely shortens the life of a sintering furnace, which is economically disadvantageous. The sintering time is preferably in a range of 10 min to 180 min.

[0080] A sintered body obtained using mixed powder for powder metallurgy according to this disclosure under the above sintering conditions through such a procedure can have higher density after sintering than the case of using alloy steel powder which does not fall within the above range even if the green
5 density is the same.

[0081] Further, the resulting sintered body may be subjected to strengthening processes such as carburized quenching, bright quenching, induction hardening, and a carbonitriding process as necessary; however, even when such strengthening processes are not performed, the sintered body using the
10 mixed powder for powder metallurgy according to this disclosure have improved strength and toughness compared with conventional sintered bodies which are not subjected to strengthening processes. The strengthening processes may be performed in accordance with conventional methods.

[0082] The disclosed iron-based sintered body obtained as described above preferably contains Mo: 0.2 mass% to 1.5 mass%, Cu: 0.5 mass% to 4.0 mass%, and C: 0.1 mass% to 1.0 mass%. Specifically, the C content is preferably in a range of 0.1 % to 1.0 % with which the highest strengthening effect and the highest fatigue strength improving effect can be achieved. When the C content is less than 0.1 %, the above advantageous effects cannot
20 be achieved. On the other hand, a C content exceeding 1.0 % results in a hypereutectoid sintered body, so that cementite is precipitated, resulting in reduced strength. Therefore, the amount of C contained in the sintered body is limited to a range of 0.1 % to 1.0 %. Preferably, the C content is 0.2 % to 0.8 %. The preferred content of Mo and Cu is determined as described above
25 for the same reasons as in the case of the above-described mixed powder for powder metallurgy.

[0083] Note that when a lubricant and the like are mixed into the above mixed powder for powder metallurgy in manufacturing a sintered body, the amount of Mo, Cu, and C in the mixed powder for powder metallurgy is controlled so
30 that the amount of Mo, Cu, and C contained the sintered body fall within the above range.

[0084] Further, the C content of the sintered body may change from the amount of graphite added depending on the sintering conditions (temperature, time, atmosphere, and others). Accordingly, when the amount of the

graphite powder added is controlled within the above range depending on the sintering conditions, an iron-based sintered body having a C content preferred in this disclosure (0.1 % to 1.0 %, more preferably 0.2 % to 0.8 %) can be manufactured.

5

EXAMPLES

[0085] A more detailed description of this disclosure will be given below with reference to examples; however, the disclosure is not limited solely to the following examples.

10 <Example 1>

As-atomized powders having particles with different circularities were used as iron-based powders. The as-atomized powders were subjected to grinding using a high speed mixer (LFS-GS-2J manufactured by Fukae Powtec Corp.) so that the circularities of the particles varied.

15 Oxidized Mo powder (mean particle diameter: 10 μ m) was added to the iron-based powders at a predetermined ratio, and the resultant powders were mixed for 15 minutes in a V blender, then subjected to heat treatment in a hydrogen atmosphere with a dew point of 30 °C (holding temperature: 880 °C, holding time: 1 h). Mo of a predetermined amount presented in Table 1
20 was then adhered to the surface of the particles of the iron-based powders by diffusion bonding to produce partially alloyed steel powders for powder metallurgy. Note that the Mo content was varied as in Samples Nos. 1 to 8 presented in Table 1.

[0086] The produced partially alloyed steel powders were each embedded into
25 a resin and polishing was performed to expose a cross section of the partially alloyed steel powder particles. Specifically, the partially alloyed steel powders were each embedded to be uniformly distributed in an area with a thickness of 0.5 mm or more in a thermosetting resin so that a cross section of a sufficient number of partially alloyed steel powder particles can be observed
30 in the polished surface, that is, the observation surface. After the polishing, the polished surface was magnified and imaged by an optical microscope, and the circularity of the particles was calculated by image analysis as described above.

Further, the specific surface area of the partially alloyed steel powder particles was measured through BET theory. The particles of each partially alloyed steel powder were confirmed to have a specific surface area of less than $0.10 \text{ m}^2/\text{g}$.

5 [0087] Subsequently, Cu powder of the mean particle diameter and the amount presented in Table 1 was added to these partially alloyed steel powders, and graphite powder (mean particle diameter: $5 \text{ }\mu\text{m}$) of the amount also presented in Table 1 was added thereto. Ethylenebisstearamide was then added in an amount of 0.6 parts by mass to the resulting mixed powder
10 for powder metallurgy: 100 parts by mass, and the powder was then mixed in a V blender for 15 minutes.

Samples Nos. 9 to 25 used partially alloyed steel powder equivalent to those used in Sample No. 5, yet the amounts of Cu powders and graphite powders varied. Samples Nos. 26 to 31 used basically the same partially
15 alloyed steel powder as that of Sample No. 5, of which mean particle diameter was adjusted by sieving. Further, Samples Nos. 32 to 38 used partially alloyed steel powders having circularities that varied.

After that, each mixed powder was compacted at a density of 7.0 g/cm^3 , thereby manufacturing bar-shaped green compacts having length: 55
20 mm, width: 10 mm, and thickness: 10 mm and ring-shaped green compacts having outer diameter: 38 mm, inner diameter: 25 mm, and thickness: 10 mm (ten pieces each). The compacting pressure was 400 MPa in each case.

[0088] The bar-shaped green compacts and the ring-shaped green compacts were sintered thereby obtaining sintered bodies. The sintering was
25 performed under a set of conditions including sintered temperature: $1130 \text{ }^\circ\text{C}$ and sintering time: 20 min in a propane converted gas atmosphere.

The measurement of outer diameter, inner diameter, and thickness and mass measurement were performed on the ring-shaped sintered bodies, thereby calculating the sintered body density (Mg/m^3). Further, the median
30 size, area fraction, and mean maximum pore length of pores in the sintered bodies were measured in accordance with the above-described methods.

For the bar-shaped sintered bodies, five of them were worked into round bar tensile test pieces (JIS No. 2), each having a parallel portion with a diameter of 5 mm, to be subjected to the tensile test according to JIS Z2241,

and the other five were bar shaped (unnotched) as sintered and had a size according to JIS Z2242 to be subjected to the Charpy impact test according to JIS Z2242. Each of these test pieces was subjected to gas carburizing at carbon potential: 0.8 mass% (holding temperature: 870 °C, holding time: 60 min) followed by quenching (60 °C, oil quenching) and tempering (holding temperature: 180 °C, holding time: 60 min).

The round bar tensile test pieces and bar-shaped test pieces for the Charpy impact test subjected to carburizing, quenching, and tempering were subjected to the tensile test according to JIS Z2241 and the Charpy impact test according to JIS Z2242; thus, the tensile strength (MPa) and the impact energy value (J/cm^2) were measured and the mean values were calculated with the number of samples $n=5$.

[0089] The measurement results are also presented in Table 1. The evaluation criteria are as follows.

(1) Flowability of mixed powder

Mixed powders for powder metallurgy: 100 g were introduced into a nozzle having diameter: 2.5 mm ϕ . When the total amount of powder was completely flown within 80 s without stopping, the powder was judged to have passed (passed). When the powder required a longer time to be flown or the total amount or part of the amount of powder stopped and failed to be flown, the powder was judged to have failed (failed).

(2) Sintered body density

A sintered body density of 6.95 Mg/m^3 or more, that is equal to or higher than that of a conventional 4Ni material (4Ni-1.5Cu-0.5Mo, maximum particle diameter of material powder: 180 μm) was judged to have passed.

(3) Tensile strength

When the round bar tensile test pieces having been subjected to carburizing, quenching, and tempering had a tensile strength of 1000 MPa or more, the test pieces were judged to have passed.

(4) Impact energy value

When the bar-shaped test pieces for the Charpy impact test having been subjected to carburizing, quenching, and tempering had an impact energy value of 14.5 J/cm^2 or more, the test pieces were judged to have passed.

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Note that the test of the impact energy value was also performed on the sintered body before carburizing, quenching, and tempering.

[0090]

Sample No.	Partially alloyed steel powder		Mo content (mass%)	Cu content (mass%)	Graphite content (mass%)	Cu particle diameter (μm)	Flowability	Sintered body								After carburizing, quenching, tempering		Evaluation	Note
	Mean particle diameter (μm)	Circularity						Mo content (mass%)	Cu content (mass%)	C content (mass%)	Pore area fraction (%)	Median pore size (μm)	Mean maximum pore size (μm)	Impact energy value (J/cm ²)	Density (N/gm ³)	Tensile strength (MPa)	Impact energy value (J/cm ²)		
1	89	0.58	0.1	2.0	0.30	35	passed	0.1	2.0	0.3	14	22	110	24	7.02	1080	13.8	failed	Comparative Example
2	91	0.60	0.2	2.0	0.30	35	passed	0.2	2.0	0.3	14	16	90	33	7.00	1125	14.7	passed	Example
3	92	0.61	0.4	2.0	0.30	35	passed	0.4	2.0	0.3	14	14	80	41	7.01	1150	15.6	passed	Example
4	95	0.62	0.6	2.0	0.30	35	passed	0.6	2.0	0.3	14	15	82	40	7.01	1175	15.4	passed	Example
5	91	0.58	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	15	16	80	39	6.97	1185	15.1	passed	Example
6	88	0.63	1.0	2.0	0.30	35	passed	1.0	2.0	0.3	14	17	87	36	6.98	1195	14.8	passed	Example
7	92	0.63	1.5	2.0	0.30	35	passed	1.5	2.0	0.3	15	17	90	34	6.95	1200	14.6	passed	Example
8	93	0.62	2.0	2.0	0.30	35	passed	2.0	2.0	0.3	15	22	110	22	6.93	1230	13.6	failed	Comparative Example
9	91	0.58	0.8	0.2	0.30	35	passed	0.8	0.2	0.3	14	21	92	23	7.01	980	13.6	failed	Comparative Example
10	91	0.58	0.8	0.5	0.30	35	passed	0.8	0.5	0.3	14	19	86	32	7.00	1015	14.6	passed	Example
11	91	0.58	0.8	1.5	0.30	35	passed	0.8	1.5	0.3	14	15	84	37	6.98	1135	15.1	passed	Example
12	91	0.58	0.8	3.0	0.30	35	passed	0.8	3.0	0.3	15	14	78	39	6.97	1210	15.4	passed	Example
13	91	0.58	0.8	4.0	0.30	35	passed	0.8	4.0	0.3	15	14	75	42	6.95	1180	15.9	passed	Example
14	91	0.58	0.8	5.0	0.30	35	passed	0.8	5.0	0.3	16	22	70	23	6.92	990	13.0	failed	Comparative Example
15	91	0.58	0.8	2.0	0.05	35	passed	0.8	2.0	0.05	14	13	73	41	7.02	980	16.0	failed	Example
16	91	0.58	0.8	2.0	0.15	35	passed	0.8	2.0	0.2	14	17	85	38	7.00	1090	15.2	passed	Example
17	91	0.58	0.8	2.0	0.50	35	passed	0.8	2.0	0.5	14	16	90	32	6.98	1150	14.8	passed	Example
18	91	0.58	0.8	2.0	1.00	35	passed	0.8	2.0	1.0	15	20	105	28	6.97	1180	14.5	passed	Example
19	91	0.58	0.8	2.0	1.50	35	passed	0.8	2.0	1.5	15	26	125	20	6.97	1115	12.0	failed	Comparative Example
20	91	0.58	0.8	2.0	0.30	55	passed	0.8	2.0	0.3	15	19	84	30	6.95	1110	14.5	passed	Example
21	91	0.58	0.8	2.0	0.30	48	passed	0.8	2.0	0.3	15	18	87	29	6.96	1140	14.6	passed	Example
22	91	0.58	0.8	2.0	0.30	30	passed	0.8	2.0	0.3	14	15	83	35	6.98	1151	15.1	passed	Example
23	91	0.58	0.8	2.0	0.30	24	passed	0.8	2.0	0.3	14	15	82	36	6.99	1160	15.1	passed	Example
24	91	0.58	0.8	2.0	0.30	15	passed	0.8	2.0	0.3	14	16	81	38	7.00	1180	15.2	passed	Example
25	91	0.58	0.8	2.0	0.30	1.5	passed	0.8	2.0	0.3	13	14	76	39	7.03	1210	15.6	passed	Example
26	128	0.48	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	16	22	100	29	6.93	995	14.0	failed	Comparative Example
27	118	0.55	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	18	82	31	6.98	1150	14.7	passed	Example
28	98	0.57	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	16	78	37	7.00	1135	15.4	passed	Example
29	75	0.58	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	15	74	42	7.01	1194	15.7	passed	Example
30	60	0.59	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	14	73	44	7.01	1230	16.0	passed	Example
31	35	0.62	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	13	72	46	6.99	1260	16.3	passed	Example
32	28	0.64	0.8	2.0	0.30	35	failed	-	-	-	-	-	-	-	-	-	-	failed	Comparative Example
33	70	0.45	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	12	71	47	7.01	1240	16.4	passed	Example
34	69	0.54	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	14	70	45	7.00	1213	16.1	passed	Example
35	72	0.56	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	14	74	43	6.99	1180	15.9	passed	Example
36	69	0.60	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	17	84	38	7.00	1140	15.0	passed	Example
37	70	0.62	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	15	19	85	35	6.97	1120	14.7	passed	Example
38	71	0.67	0.8	2.0	0.30	35	passed	0.8	2.0	0.3	14	28	100	19	6.98	1001	12.0	failed	Comparative Example
39*	65	0.67	0.5	-	0.30	35	passed	0.5	1.5	0.3	15	29	130	25	6.97	998	13.3	failed	Comparative Example

Sample No. 39 is a 4Ni material (Fe-4Ni-1.5Cu-0.5Mo)

[0091] For each of Samples Nos. 1, 8, 9, 14, 19, 26, 38, and 39*, the median size D50 of the pores in the sintered body exceeded 20 μm , resulting in a low impact energy value, lack of toughness, and reduced tensile strength.

Further, for comparing the effects of the components in the sintered bodies, the Mo content in Sample Nos. 1 to 8, the Cu content in Nos. 9 to 14, and the graphite content in Nos. 15 to 19 were contrasted. Similarly, Samples Nos. 20 to 25 were designed for evaluating the effect of the Cu particle diameter, Nos. 26 to 31 for evaluating the effect of the alloyed particle diameter, and Nos. 32 to 38 for evaluating the effect of the circularity and the mean particle diameter of the partially alloyed steel powders. Table 1 also presents the results of a 4Ni material (4Ni-1.5Cu-0.5Mo, maximum particle diameter of material powder: 180 μm) as the conventional material. The table demonstrates that our examples exhibited better properties over the conventional 4Ni material.

As presented in Table 1, all of Examples of this disclosure were sintered bodies having high tensile strength and toughness.

[0092] <Example 2>

Three atomized iron powders having particles of different specific surface areas and circularities were prepared. The specific surface area and the circularity were adjusted by grinding each atomized iron powder using a high speed mixer (LFS-GS-2J manufactured by Fukae Powtec Corp.) and adjusting the mixing ratio of coarse powder having a particle size of 100 μm or more and fine powder having a particle size of 45 μm or less.

[0093] Oxidized Mo powder (mean particle diameter: 10 μm) was added to the iron-based powders at a predetermined ratio, and the resultant powders were mixed for 15 minutes in a V blender, then subjected to heat treatment in a hydrogen atmosphere with a dew point of 30 $^{\circ}\text{C}$ (holding temperature: 880 $^{\circ}\text{C}$, holding time: 1 h). Mo of a predetermined amount presented in Table 2 was then adhered to the surface of the particles of the iron-based powders by diffusion bonding to produce partially alloyed steel powders for powder metallurgy. These partially alloyed steel powders were each embedded into a resin and polishing was performed to expose a cross section of the partially alloyed steel powder particles. Subsequently, the cross section was magnified and imaged by an optical microscope, and the circularity of the

particles was calculated by image analysis. Further, the specific surface area of the partially alloyed steel powder particles was measured through BET theory.

[0094] Next, 2 mass% of Cu powder having a mean particle diameter of 35 μm was added to these partially alloyed steel powders, and 0.3 mass% of graphite powder (mean particle diameter: 5 μm) was added thereto. Ethylenebisstearamide was then added in an amount of 0.6 parts by mass to the resulting mixed powder for powder metallurgy: 100 parts by mass, and the powder was then mixed in a V blender for 15 minutes. Each of the mixed powders was compacted at a compacting pressure of 686 MPa, thereby manufacturing bar-shaped green compacts having length: 55 mm, width: 10 mm, and thickness: 10 mm and ring-shaped green compacts having outer diameter: 38 mm, inner diameter: 25 mm, and thickness: 10 mm (ten pieces each).

[0095] The bar-shaped green compacts and ring-shape green compacts were sintered to obtain sintered bodies. The sintering was performed under a set of conditions including sintered temperature: 1130 °C and sintering time: 20 min in a propane converted gas atmosphere.

The measurement of outer diameter, inner diameter, and thickness and mass measurement were performed on the ring-shaped sintered bodies, thereby calculating the sintered body density (Mg/m^3). Further, the median size, area fraction, and mean maximum pore length of pores in the sintered bodies were measured in accordance with the above-described methods.

[0096] For the bar-shaped sintered bodies, five of them were worked into round bar tensile test pieces (JIS No. 2) having diameter: 5 mm to be subjected to the tensile test according to JIS Z2241, and the other five were bar shaped (unnotched) as sintered to be subjected to the Charpy impact test according to JIS Z2242. Each of these test pieces was subjected to gas carburizing at carbon potential: 0.8 mass% (holding temperature: 870 °C, holding time: 60 min) followed by quenching (60 °C, oil quenching) and tempering (holding temperature: 180 °C, holding time: 60 min).

The round bar tensile test pieces and bar-shaped test pieces for the Charpy impact test subjected to carburizing, quenching, and tempering were subjected to the tensile test according to JIS Z2241 and the Charpy impact test

according to JIS Z2242; thus, the tensile strength (MPa) and the impact energy value (J/cm^2) were measured and the mean values were calculated with the number of samples $n=5$.

The measurement results are also presented in Table 2. The
5 acceptance criteria for the values of the properties were the same as those in Example 1.

[0097]

Table 2]

Sample No.	Partially alloyed steel powder			Mo content (mass%)	Cu content (mass%)	Graphite content (mass%)	Cu particle diameter (μm)	Flowability	Sintered body							After carburizing, quenching, tempering		Note		
	Mean particle diameter (μm)	Circularity	Specific surface area (m ² /g)						Mo content (mass%)	Cu content (mass%)	C content (mass%)	Pore area fraction (%)	Median pore size (μm)	Maximum pore length (μm)	Impact energy value (J/cm ²)	Density (Mg/m ³)	Tensile strength (MPa)		Impact energy value (J/cm ²)	
40	78	0.55	0.07	0.4	2.0	0.3	35	passed	0.4	2.0	0.3	14	16	85.0	35.0	7.01	1175	15.1	passed	Example
41	76	0.52	0.08	0.8	2.0	0.3	35	passed	0.8	2.0	0.3	15	14	73.0	42.0	6.97	1194	15.7	passed	Example
42	76	0.59	0.13	0.4	2.0	0.3	35	-	0.4	2.0	0.3	-	-	-	-	-	-	-	failed	Comparative Example
43	77	0.52	0.15	0.8	2.0	0.3	35	-	0.8	2.0	0.3	-	-	-	-	-	-	-	failed	Comparative Example
44	76	0.67	0.12	0.4	2.0	0.3	35	-	0.4	2.0	0.3	-	-	-	-	-	-	-	failed	Comparative Example
45	77	0.66	0.14	0.8	2.0	0.3	35	-	-	-	-	-	-	-	-	-	-	-	failed	Comparative Example
46	75	0.68	0.06	0.4	2.0	0.3	35	passed	0.4	2.0	0.3	12	25	110.0	21.0	7.10	1060	12.1	failed	Comparative Example
47	77	0.69	0.08	0.8	2.0	0.3	35	passed	0.8	2.0	0.3	13	25	100.0	20.0	7.06	1075	12.3	failed	Comparative Example

[0098] As can be seen from Table 2, all the sintered bodies having a median pore size D50 of 20 μm or less had a high impact energy value, excellent toughness, and high tensile strength. Further, when partially alloyed steel powders having particles of a circularity and a specific surface area within the disclosed range were used, the target values of the sintered body density, the tensile strength, and the impact energy value were achieved.

CLAIMS

1. An iron-based sintered body, comprising:
partially diffusion alloyed steel powder in which Mo is adhered to the surface of particles of iron-based powder;
Cu powder; and
graphite powder; wherein
the partially diffusion alloyed steel powder has a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m^2/g , and a circularity of particles of the partially diffusion alloyed steel powder that have a diameter in a range of 50 μm to 100 μm is 0.65 or less,
the iron-based sintered body has a chemical composition consisting of:
Mo in an amount of 0.2 mass% to 1.5 mass%,
Cu in an amount of 0.5 mass% to 4.0 mass%,
C in an amount of 0.1 mass% to 1.0 mass%, and
the balance consisting of Fe and incidental impurities, and
the iron-based sintered body has an area fraction of pores in the iron-based sintered body more than 0% and of 15 % or less, and an area-based median size D50 of the pores of 20 μm or less.
2. The iron-based sintered body according to claim 1, wherein the iron-based sintered body has been carburized, quenched, and tempered.
3. A method of manufacturing an iron-based sintered body, the method comprising:
(a) a step of diffusion bonding of iron-based powder with Mo to obtain partially diffusion alloyed steel powder in which Mo is adhered to the surface of particles of iron-based powder;
(b) a step of mixing at least Cu powder and graphite powder with the partially diffusion alloyed steel powder to obtain mixed powder for powder metallurgy;
(c) a step of compacting the mixed powder for powder metallurgy at a pressure of 400 MPa or more to obtain a compact; and
(d) then sintering the obtained compact at 1000 $^{\circ}\text{C}$ or higher for 10 min or more;

wherein

the partially diffusion alloyed steel powder has a mean particle diameter of 30 μm to 120 μm and a specific surface area of less than 0.10 m^2/g , and a circularity of particles of the partially diffusion alloyed steel powder that have a diameter in a range of 50 μm to 100 μm is 0.65 or less, and

the obtained iron-based sintered body has a chemical composition consisting of:

Mo in an amount of 0.2 mass% to 1.5 mass%,

Cu in an amount of 0.5 mass% to 4.0 mass%,

C in an amount of 0.1 mass% to 1.0 mass%, and

the balance consisting of Fe and incidental impurities.

4. The method of manufacturing an iron-based sintered body according to claim 3, the method further comprising carburizing, quenching, and tempering after sintering the obtained compact.