(11) EP 1 985 393 A1

(12)

EUROPEAN PATENT APPLICATION published in accordance with Art. 158(3) EPC

(43) Date of publication:

29.10.2008 Bulletin 2008/44

(21) Application number: 07714625.6

(22) Date of filing: 14.02.2007

(51) Int Cl.:

B22F 3/02^(2006.01) C22C 33/02^(2006.01) B22F 1/00 (2006.01) C22C 38/00 (2006.01)

(86) International application number:

PCT/JP2007/053125

(87) International publication number:

WO 2007/105429 (20.09.2007 Gazette 2007/38)

(84) Designated Contracting States: **DE SE**

(30) Priority: 15.02.2006 JP 2006037916

15.12.2006 JP 2006337876

(71) Applicant: JFE Steel Corporation

Tokyo, 100-0011 (JP)

(72) Inventors:

 OZAKI, Yukiko Tokyo 100-0011 (JP) ONO, Tomoshige

Tokyo 100-0011 (JP)

 UNAMI, Shigeru Tokyo 100-0011 (JP)

• FUJINAGA, Masashi

Tokyo 100-0011 (JP)

(74) Representative: Grünecker, Kinkeldey,

Stockmair & Schwanhäusser

Anwaltssozietät Leopoldstrasse 4

80802 München (DE)

(54) IRON-BASE MIXED POWDERS AND PROCESSES FOR PRODUCTION OF IRON-BASE POWDER COMPACTS AND SINTERED IRON-BASE POWDER COMPACTS

(57) An iron-based powder mixture for powder metallurgy is provided, in which iron-based powder is blended with at least one selected from talc and steatite, and preferably further blended with metallic soap, thereby when a compacted body is sintered, furnace environment

is not adversely affected, and excellent compaction performance is achieved even in a low temperature range of less than 100°C, and more preferably, an obtained sintered body has excellent machining performance.

EP 1 985 393 A1

Description

Technical Field

[0001] The present invention relates to an iron-based powder mixture including iron-based power mixed with a lubricant, and alloying powder as needed. The iron-based powder mixture of the invention is suitable for powder metallurgy, and particularly suitable for compaction in a temperature range from normal temperature to less than 100°C.

[0002] The invention further relates to a powder mixture for powder metallurgy, which is preferable for manufacturing of high-strength sintered parts for automobiles.

[0003] Moreover, the invention relates to a method of manufacturing an iron-based compacted body using the iron-based powder mixture as a material, and a method of manufacturing an iron-based power sintered-body using the iron-based compacted body as a material.

Background Art

15

20

30

35

40

45

50

55

[0004] The iron-based powder mixture for powder metallurgy is typically manufactured in a way that iron-based powder is added with a lubricant and alloying powder and mixed, and furthermore added with powder of free machining additives and mixed as needed.

[0005] Here, the iron-based powder is a main component of the powder mixture, and iron powder (including pure iron powder), or alloyed steel powder is mainly used as the iron-based powder. The alloyed steel powder contains an alloyed element. While steel powder containing no C may be used as the alloyed steel powder, steel powder containing C and iron powder containing no C are generally called alloyed steel powder herein. In addition to the above, partly diffused alloyed steel powder may be used, in which an alloy element is bonded to pure iron powder or the like by partial diffusion. In the application, the partly diffused alloyed steel powder is assumed to be a type of the alloyed iron powder.

[0006] The lubricant is an additive that is added particularly for facilitating compaction or ejection of a compacted body from a die after compaction. While various substances can be used for the lubricant, the lubricant is selected in consideration of a mixing property with iron-based powder or a decomposition property during sintering. As an example of the lubricant, zinc stearate, aluminum stearate, lead stearate and the like are listed. Various lubricants are exemplified in US Patent No.5,256,185 and the like.

[0007] The alloying powder is added mainly for adjusting a composition and/or a structure of an iron-based compacted body or an iron-based sintered body, and includes graphite powder, copper powder, iron phosphide powder, molybdenum powder, and nickel powder.

[0008] The powder of free machining additives (or free machining elements), such as S or MnS, is added particularly for improving machining performance of the sintered body.

[0009] Recently, with increase in demand for increasing strength of sintered parts, as disclosed in Japanese Unexamined Patent Application Publication JP-A-2-156002 (1990), Japanese Examined Patent Application Publication JP-B-7-103404 (1995), and US Patent No.5,368,630, a warm compaction technique has been developed, in which an iron-based powder mixture is compacted while being heated, thereby increase in density and increase in strength of a compacted body can be achieved. According to the technique, density of a compacted body can be increased at a relatively low load by using a phenomenon that iron-based powder is gradually reduced in resistance to plastic deformation as the powder is heated.

[0010] However, such an iron-based powder mixture has the following problems. That is, the warm compaction is a technique that a die and powder are heated to high temperature beforehand, then the iron-based powder mixture is compacted. As the heating temperature, while a range of 70 to 120°C is described in JP-A-2-156002, heating is substantially preferably performed at 100°C or more as described in JP-B-7-103404 and USP 5,368,630. However, since it is very difficult that the iron-based powder mixture having low heat conductivity is uniformly heated to 100°C or more, and kept at the temperature, productivity of sintered parts have been likely reduced. Moreover, the iron-based powder mixture is heated for a long time, resulting in a problem that the iron-based powder mixture is oxidized.

[0011] JP-A-9-104901 (1997) or JP-A-10-317001 (1998) discloses a technique that an inorganic compound having a layered crystal such as MoS_2 , carbon fluoride, and graphite is used as the lubricant. However, when MoS_2 is used, the MoS_2 may be decomposed during sintering, causing generation of harmful sulfur gas that possibly contaminates a furnace. When carbon fluoride is used so that the iron-based powder mixture is sintered in a hydrogen atmosphere, there is fear that corrosive hydrogen fluoride may be generated.

[0012] Therefore, it is desired to develop an iron-based powder mixture having high compressibility similar to that of a warm-rolled, iron-based powder mixture, even if it is not subjected to warm rolling.

[0013] On the other hand, for the iron-based powder mixture, the problem of machining performance is also desired to be solved

[0014] When parts of various machines such as automobiles are manufactured by a powder metallurgy technique, a

powder mixture for powder metallurgy is filled in a die and compacted, and furthermore sintered. Parts of various machines obtained in this way (hereinafter, called sintered parts) typically have a density of 5.0 to 7.2 g/cm³ respectively. Moreover, since each of the sintered parts is good in dimension accuracy, a part having a complicated shape can be produced.

[0015] The sintered parts are used for parts of various machines. In particular, parts for automobiles (for example, gears) are required to have high strength and high fatigue characteristics. Thus, a technique of using a powder mixture for powder metallurgy, which is added with an alloyed element, is variously investigated in order to manufacture a sintered part having high strength and high fatigue characteristics. For example, JP-B-45-9649 (1970) discloses a powder mixture for powder metallurgy, which includes pure Fe powder diffusion-bonded with powder of Ni, Cu, Mo or the like, and is preferable for manufacturing a sintered part having high strength and high fatigue characteristics, and is excellent in compressibility. Moreover, as a powder mixture for powder metallurgy preferable for manufacturing a sintered part having high strength, JP-A-61-163239 (1986) discloses a powder mixture for powder metallurgy, which includes low alloyed-steel powder, in which C and Mo are contained, and Mn and Cr are substantially not contained, the steel powder being added with Cu powder and/or Ni powder, and furthermore, added with graphite powder. Moreover, JP-A-63-114903 (1988) discloses a powder mixture for powder metallurgy, in which Cu powder is diffusion-bonded to alloyed steel powder containing Mo, Mn and C.

[0016] However, even if powder metallurgy techniques are used, when a sintered part, which is required to have extremely strict dimension accuracy, is manufactured, the sintered part needs to be subjected to machining (such as cutting or drilling) after sintering. However, since a sintered part is bad in machining performance, a cutting tool used in the machining is significantly worn. As a result, machining cost is increased, leading to increase in manufacturing cost of a sintered part. Such degradation in machining performance of a sintered part is caused by a phenomenon that a solid surface intermittently appears in the inside of a work material due to pores within the sintered part, which intermittently give a shock to a tool during cutting, in addition, heat conductivity of the sintered part is thus decreased, and consequently temperature of the sintered part is increased during cutting. The machining performance is significantly degraded as strength of a sintered part is increased.

[0017] As described before, it is previously known that the powder mixture for powder metallurgy is added with free machining additives, thereby machining performance of a sintered part is improved. The free machining additives have an effect of easily breaking chips, or an effect of forming a thin built-up edge on a surface of a cutting tool to improve lubricity of the cutting tool (particularly, on a rake face).

[0018] However, free machining additives containing S as a main component, like MoS₂ described above, contaminate a furnace. Moreover, in the techniques disclosed in JP-B-45-9649, JP-A-61-163239, and JP-A-63-114903, since hardness of the obtained sintered part is particularly high, even if the free machining additives are added to the powder mixture for powder metallurgy, significant improvement of machining performance cannot be expected.

[0019] As a technique of eliminating a bad effect on the furnace to improve machining performance of a sintered part, a technique of using an MgO-SiO₂ composite oxide is proposed. For example, JP-A-1-255604 (1989) discloses a technique that an MgO-SiO₂ composite oxide (for example, anhydrous talc), in which MgO/SiO₂ is 0.5 or more and less than 1.0 in mol ratio, and crystallization water is not contained, is blended to iron-based powder as means of improving machining performance without reducing mechanical properties (for example, strength) of a sintered body. Moreover, JP-A-64-79302 (1989) discloses a technique that free machining additives including a MgO-SiO₂ composite oxide and/or glass powder are contained in reduced iron powder in a configuration that the additives stay inside of each iron powder particle (that is, the additives are added to iron powder raw material before reduction).

[0020] Any of the publications describes that the composite oxide is preferably added in a range of 0.1 to 1.5 wt%. However, according to a result of investigation on iron-based powder containing a lubricant (zinc stearate of 1 wt%) or the like, as an added amount of the composite oxide is increased, an effect of improving machining performance is increased, and particularly large effect is obtained in a range of 0.5 to 1.0 wt%, but on the other hand, mechanical properties are reduced (Table 3 in JP-A-1-255604, and Figs 6 and 8 in JP-A-64-79302). That is, the techniques are not necessarily advantageous in a point of quality of a sintered body.

Disclosure of the Invention

20

30

35

40

45

55

50 Problems that the Invention is to Solve

[0021] The invention advantageously solves the problems, and an object of the invention is to propose an iron-based powder mixture for powder metallurgy, which has no adverse effect on furnace environment during sintering a compact, and provides excellent compaction performance that the powder mixture can be compacted at high density even in a low temperature region of less than 100°C.

[0022] Moreover, in consideration of increase in demand for improving machining performance of a sintered part to reduce machining cost, another object of the invention is to provide an iron-based powder mixture for powder metallurgy preferable for machining a sintered part having excellent machining performance, and particularly preferable for machining performance.

chining a high-strength sintered part.

[0023] Still another object of the invention is to propose a method of manufacturing an iron-based compacted body using the iron-based powder mixture as a material, and furthermore, a method of manufacturing an iron-based sintered body using the iron-based compacted body as a material.

Means for Solving the Problems

5

20

25

30

35

40

45

50

55

[0024] As a measure for solving the problems, the inventors made earnest investigations on a particular lubricant, by which when an iron-based powder mixture is compacted, furnace environment is not adversely affected, and even if the iron-based powder mixture is compacted at a relatively low heating temperature of the iron-based powder mixture, and preferably even if it is compacted without being heated, a high-density compacted body can be manufactured.

[0025] As a result, they had a finding that when talc or steatite was used as a lubricant, and furthermore, fatty acid amide was used, rearrangement of iron-based powder particles was accelerated during compaction, consequently even if compaction temperature was low, that is, about room temperature, an iron-based compacted body having high compaction density was obtained.

[0026] Moreover, it was found that when metallic soap was added, an extremely high effect of improving machining performance was obtained by talc or steatite in a low added amount compared with a previously known amount, which had no adverse influence on mechanical properties.

[0027] The invention is designed based on the above findings.

[0028] That is, summary and a configuration of the invention are as follows.

- (1) An iron-based powder mixture, characterized by containing iron-based powder, and containing as additives;
 - at least one selected from talc and steatite, and
 - fatty acid amide.
- (2) The iron-based powder mixture according to the above (1), characterized in that the additives further contain metallic soap.
- (3) The iron-based powder mixture according to the above (1) or (2), characterized in that the iron-based powder mixture is further blended with alloying powder.
- (4) The iron-based powder mixture according to the above (3), characterized in that the iron-based powder is water-atomized alloyed steel powder containing Mo of 0.3 to 0.5 mass%, Mn of 0.1 to 0.25 mass%, and the remainder being Fe and inevitable impurities, and the alloying powder is Cu powder of 1 to 3 mass% and graphite powder of 0.5 to 1.0 mass%.
- (5) An iron-based powder mixture, characterized in that the iron-based powder mixture is formed by mixing water-atomized alloyed steel powder containing Mo of 0.3 to 0.5 mass%, Mn of 0.1 to 0.25 mass%, and the remainder being Fe and inevitable impurities, Cu powder of 1 to 3 mass%, graphite powder of 0.5 to 1.0 mass%, at least one selected from talc and steatite in a range of 0.05 to 0.5 mass% in total, and fatty acid amide.
- (6) The iron-based powder mixture according to the above (5), wherein the iron-based powder mixture further contains metallic soap.
- (7) A method of manufacturing an iron-based compacted body, characterized in that the iron-based powder mixture according to any one of the above (1) to (6) is filled in a die, and then compacted at a temperature of less than 100°C.
- (8) A method of manufacturing an iron-based sintered body, characterized in that the iron-based powder mixture according to any one of the above (1) to (6) is filled in a die, then compacted at a temperature of less than 100°C, and then an obtained iron-based compacted body is sintered.

[0029] Each of the content of an alloyed element (including Mo or Mn) in the iron-based powder, the amount of alloying powder (including Cu powder and graphite powder) to be added, and the added amount of talc or steatite refers to percentage of mass of the iron-based powder mixture.

Best mode for Carrying Out the Invention

[0030] Hereinafter, the invention is specifically described.

[0031] First, materials of the iron-based powder mixture of the invention are described. The content of each of alloyed elements in the iron-based powder, and the blending amount of each of the materials (alloying powder, lubricant and the like) are expressed in a weight percent of mass (100 mass%) of an iron-based powder mixture obtained by mixing those, that is, the weight percent is expressed using a numerical value included in a numerical value of the mass of the powder mixture. However, such a weight percent is not significantly different in numerical value from that in the case

that the alloy content (including the amount of partly diffused alloy) and the like in the iron-based powder is expressed in a weight percent of mass of the iron-based powder.

<Iron-based powder>

5

[0032] In the invention, as the iron-based powder, pure iron powder such as atomized iron powder or reduced iron powder, or alloyed steel powder is exemplified. As the alloyed steel powder, partly-diffused alloyed steel powder and prealloyed steel powder (in which alloyed elements are already contained when melted) are exemplified, and furthermore, hybrid steel powder is exemplified, in which alloyed elements are partly diffused in the prealloyed steel powder.

[0033] The content of impurities in the iron-based powder may be about 3 mass% or less in total. The content of each of typical impurities is as follows: C is 0.05 mass% or less, Si is 0.10 mass% or less, Mn (in the case that Mn is not added as an alloy element) is 0.50 mass% or less, P is 0.03 mass% or less, S is 0.03 mass% or less, O is 0.30 mass% or less, and N is 0.1 mass% or less.

[0034] For the alloyed steel powder, Cr, Mn, Ni, Mo, V, Ti, Cu, Nb and the like can be alloyed. In particular, Ti, Ni, Mo, Cu and the like can be added even by diffusion bonding. If the precondition as the iron-based powder (Fe content is 50 mass% or more) is satisfied, other alloy elements are not particularly limited in content.

[0035] Average particle diameter of the iron-based powder is preferably adjusted to be in a typically used range for powder metallurgy, that is, in a range of about 70 to 100 μ m. The particle diameter of the powder is shown as a measurement value by a sieving method according to JIS Z 2510, unless otherwise specified.

[0036] Hereinafter, a specific composition of alloyed steel powder particularly preferable for a material of a high-strength sintered body is exemplified.

(Iron-based powder example 1)

[0037] As a first example, prealloyed steel powder is preferably shown, which contains Mo of 0.3 to 0.5 mass%, Mn of 0.1 to 0.25 mass%, and the remainder being Fe and inevitable impurities. In the light of productivity, the steel powder is preferably water-atomized alloyed steel powder, which is manufactured by water-atomizing the steel having the above composition.

[0038] The reason for a preferable range of each component is as follows.

30

20

• Mo: 0.3 to 0.5 mass%

35

[0039] Mo is an element that increases strength of a sintered part by solution hardening or improvement in hardenability (quench hardenability) of alloyed steel powder. When Mo content is less than 0.3 mass%, an effect of increasing strength of the sintered part by Mo is not obtained. On the other hand, when the content is more than 0.5 mass%, since the effect of increasing strength of the sintered part is saturated, machining performance is unnecessarily reduced. Therefore, Mo content is preferably adjusted to be in a range of 0.3 to 0.5 mass%.

• Mn: 0.1 to 0.25 mass%

40

45

50

[0040] Mn is an element that increases strength of a sintered part by solution hardening or improvement in hardenability of water-atomized alloyed steel powder. When Mn content is less than 0.1 mass%, an effect of increasing strength of the sintered part by Mn is not obtained. On the other hand, when the content is more than 0.25 mass%, oxidation of Mn easily proceeds, leading to reduction in strength and compressibility of alloyed steel powder. Therefore, Mn content is preferably adjusted to be in a range of 0.1 to 0.25 mass%.

[0041] The rest of the powder other than the above components preferably is Fe and inevitable impurities. The inevitable impurities inevitably gets into the steel in a stage that an ingot being a material of the water-atomized alloyed steel powder is produced, or in a stage that water-atomized alloyed steel powder is manufactured from the ingot.

[0042] A preferable method of manufacturing the water-atomized alloyed steel powder is described, the method being preferably used in the invention. An ingot containing a predetermined composition (that is, the above composition) is produced, and then the ingot is formed into powder by a water atomizing method. Furthermore, the obtained powder is subjected to finish reduction and crushing (or pulverizing) thereby obtaining water-atomized alloyed steel powder. An apparatus for obtaining powder from an ingot by the water atomizing method is not limited to a particular type, and any previously known apparatus may be used as the apparatus.

55

<Alloying powder>

[0043] As the alloying powder, graphite powder, metal powder of such as Cu, Mo and Ni, boron powder, cuprous oxide

powder and the like are exemplified. Such alloying powder is mixed to the iron-based powder, so that strength of a sintered body can be increased.

[0044] The blending amount of the alloying powder is preferably adjusted to be about 0.1 to 10 mass% in the iron-based powder mixture. The reason for this is that the alloying powder is blended by 0.1 mass% or more, so that strength of an obtained sintered body is advantageously improved, on the other hand, when it is blended by more than 10 mass%, dimension accuracy of the sintered body is reduced.

[0045] In the case of the iron-based powder example 1, particularly Cu powder of 1 to 3 mass% and graphite powder of 0.5 to 1.0 mass% are preferably added.

[0046] C being a main component of graphite powder is an element that increases strength of a sintered part by solution hardening or improvement in hardenability of water-atomized alloyed steel powder. When the added amount of graphite powder is less than 0.5 mass%, a desired effect is not sufficiently obtained in the iron-based powder example 1. On the other hand, when the content is more than 1.0 mass%, strength of the sintered part is increased beyond necessity, and consequently machining performance is unnecessarily reduced. Therefore, the content of graphite powder is adjusted to be in a range of 0.5 to 1.0 mass%.

[0047] Cu is an element that increases strength of a sintered part by solution hardening or improvement in hardenability of alloyed steel powder. Moreover, Cu powder is melted during sintering and thus changed into a liquid phase, causing adhesion of particles of the alloyed steel powder to one another. When the added amount of Cu powder is less than 1 mass%, a desired effect is not sufficiently obtained in the iron-based powder example 1. On the other hand, when the amount is more than 3 mass%, since the effect of increasing strength of the sintered part is saturated, machining performance is unnecessarily reduced. Therefore, the content of Cu powder is adjusted to be in a range of 1 to 3 mass%. [0048] When Cu powder is added, if the added amount is within the above range, an adding method may be a method where alloyed steel powder is added with Cu powder and then simply mixed, or a method of adhering Cu powder on a surface of water-atomized alloyed steel powder via a binder. Moreover, it is acceptable that the alloyed steel powder and the Cu powder is diffusion-bonded on a surface of the alloyed steel powder so as to be formed into partly-diffused alloyed steel powder (or hybrid alloyed steel powder).

<Talc/steatite>

20

30

35

40

45

50

55

[0049] In the invention, it is important that at least one selected from talc (3MgO-4SiO₂) and steatite is blended. Steatite is sometimes called fired talc, and contains enstatite (MgO-SiO₂) as a main component.

[0050] When talc or steatite is added together with fatty acid amide, it exhibits a particularly large effect as a lubricant. Moreover, while talc or steatite is one of MgO-SiO₂ composite oxides known as free machining additives, if talc or steatite is further added together with metallic soap, it exhibits a particularly large effect even as a free machining additive.

[0051] The talc or steatite is blended as the lubricant, thereby compressibility of a compacted body is improved, in addition, ejection force in compaction process is reduced, so that compaction performance is remarkably improved. The reason for this is considered as follows.

[0052] That is, it is considered that when talc, steatite, and boron nitride are subjected to shear stress between iron-based powder particles during compaction, each of the substances tends to be cleaved along a crystal face, therefore frictional resistance between particles within a compacted body is reduced, and thus the particles easily move with respect to each other, as a result, density of the compacted body is improved. Such an effect is effective in a region of a relatively low compressive stress. On the other hand, in a high pressure region, fatty acid amide exhibits an effect that it thinly enters into a space between the particles so as to reduce frictional resistance. It is considered that since the frictional resistance is reduced over all the compressive regions in this way, a synergetic effect is exhibited for increasing density of the compacted body.

[0053] Moreover, it is considered that when talc or steatite exists between a compacted body and a die, since the talc or steatite is cleaved due to shear stress applied from a die surface during ejecting the compacted body, slidability of the compacted body on the die surface is improved, leading to reduction in ejection force.

[0054] Since the effects are exhibited regardless of temperature of an iron-based powder mixture, the iron-based powder mixture is not necessarily heated, and the effects effectively contribute to increasing density of an iron-based compacted body in compaction even at normal temperature. Moreover, when the iron-based powder is heated, since plastic deformation resistance of the iron-based powder is decreased during compaction, higher density of a compacted body can be obtained. Therefore, while heating temperature of the iron-based powder can be appropriately set depending on a required density of a compacted body, sufficient heating temperature is less than 100°C. More preferably, the heating temperature is 80°C or less.

[0055] While the reason why machining performance is remarkably improved is not elucidated, it is possibly considered that a metal component in metallic soap reacts with talc/steatite during sintering, so that the metal component acts as an auxiliary free machining additive. A sintered part manufactured by using the powder mixture for powder metallurgy of the invention may have high strength similar to that of a usual high-strength sintered part, and in addition, may have

extremely excellent machining performance.

[0056] The blending amount of the talc or steatite is preferably adjusted to be about 0.01 to 0.5 mass% in total in the iron-based powder mixture. The reason for this is that such a lubricant blended by 0.01 mass% or more, thereby density of a compacted body can be adequately increased during compaction, and ejection force can be adequately decreased during ejecting the compacted body. Moreover, when an effect of improving machining performance is intended to be obtained, the lubricant is preferably added by 0.01 mass% or more, too. When alloyed steel powder for a high-strength sintered body (for example, the iron-based powder example 1) is used, to secure a stronger effect of improving machining performance, the added amount of talc and/or steatite is preferably adjusted to be 0.05 mass% or more in total.

[0057] On the other hand, when the blending amount is more than 0.5 mass% or more, compressibility of the powder mixture is reduced, which may reduce mechanical strength and the like of a sintered body obtained by sintering the compacted body. More preferably, an upper limit of the blending amount is 0.3 mass%, and the upper limit is preferably adjusted to be 0.2 mass% or less to substantially eliminate influence on mechanical properties of the sintered body.

[0058] Preferably, talc has a monoclinic or triclinic crystal structure, steatite has a monoclinic crystal structure, and boron nitride has a hexagonal crystal structure.

15 [0059] Size of talc or steatite is preferably about 1 to 10 μ m in particle diameter.

<Fatty acid amide>

[0060] In the invention, at least one of fatty acid amides is blended as a lubricant. Here, as the fatty acid amide, at least one selected from fatty acid monoamide (such as stearic acid monoamide), and fatty acid bisamide (such as ethylene-bis-stearoamide and methylene-bis-stearoamide) is preferably used.

[0061] Each of them acts as not only a lubricant, but also a binder. Therefore, by using each of them, segregation or dusting of the relevant iron-based powder mixture is effectively prevented, and flowability and compaction performance can be further improved. While a fatty acid is sometimes mixed in fatty acid amide, this is not particularly prohibited.

[0062] The blending amount of the fatty acid amide is preferably adjusted to be about 0.01 to 0.5 mass% in the iron-based powder mixture. The reason for this is that when the blending amount is less than 0.01 mass%, the adding effect is poor, and on the other hand, when the blending amount is more than 0.5 mass%, strength of a compacted body (or green compact) is decreased. A lower limit of the blending amount is more preferably 0.03 mass% in the case that the iron-based powder is pure iron powder, and 0.05 mass% in the case that it is alloyed steel powder. An upper limit of the blending amount is more preferably 0.4 mass%, and in the case that the iron-based powder is pure iron powder, 0.3 mass% is further more preferable as the upper limit.

<Metallic soap>

30

40

45

55

[0063] In the invention, metallic soap can be further blended. According to a previous common idea, the metallic soap is also counted as a lubricant.

[0064] As the metallic soap, zinc stearate, lithium stearate, calcium stearate and the like are listed. Among them, the zinc stearate and the lithium stearate are particularly preferable.

[0065] The blending amount of the metallic soap is preferably adjusted to be about 0.01 to 0.5 mass% in the iron-based powder mixture. The reason for this is that when the blending amount is less than 0.01 mass%, the adding effect is poor, and on the other hand, when the blending amount is more than 0.5 mass%, strength of a compacted body is decreased. A lower limit of the blending amount is more preferably 0.05 mass% or more, and an upper limit thereof is more preferably 0.3 mass%.

[0066] The added amount of the fatty acid amide and the metallic soap in total is preferably adjusted to be 0.1 mass% to 1.0 mass%. The lower limit is more preferably 0.2 mass%, and the upper limit is more preferably 0.6 mass%.

[0067] Furthermore, the blending amount of the talc/steatite, the fatty acid amide, and the metallic soap in total is preferably adjusted to be about 0.01 to 2.0 mass% in the iron-based powder mixture. The lower limit is more preferably 0.15 mass%, and the upper limit is more preferably 0.8 mass%.

50 <Other materials>

[0068] While other additives are not particularly needed for the iron-based powder mixture of the invention, a known additive such as surface modification agent (including siloxanes) may be further added by about 0.5 mass% or less.

<Method of manufacturing powder mixture>

[0069] Next, a method of manufacturing the iron-based powder mixture of the invention is described.

(First method)

[0070] Iron-based powder is added with the respective materials (such as talc, steatite, fatty acid amide, metallic soap, and alloying powder), and then subjected to primary mixing. Then, a mixture after primary mixing is agitated while it is heated to a melting point or higher of at least one of the fatty acid amide and metallic soap, and then the mixture is gradually cooled while being mixed. As a result, the alloying powder or other material powder is adhered on the iron-based powder by an effect of the melted material.

[0071] That is, the material, which is melted and used for adhesion, acts even as a binder.

(Second method)

20

30

35

45

55

[0072] As a method similar to the first method, it is also possible that the iron-based powder is added with only some of the materials, and subjected to primary mixing, and then further added with the rest of the materials, and subjected to secondary mixing. The material subjected to secondary mixing exists in the powder mixture in a free state. As a particularly preferable example, a method is given, in which at least part of the metallic soap is supplied for the secondary mixing, and the rest of the materials is supplied for the primary mixing, and fatty acid amide, or a co-melt of the fatty acid amide with the metallic soap is used for the binder. According to the method, the added amount of each material to be blended to the iron-based powder can be minimized.

[0073] Mixing means of the iron-based powder and each material is not particularly limited, and any of previously known mixers can be used. In particular, a high-speed mixer, counter current mixer, plough share mixer, and conical mixer, in each of which the material powders being easily heated, are particularly advantageously suited.

<Method of manufacturing compacted body and sintered body>

[0074] Next, a method of manufacturing an iron-based compacted body using the iron-based powder mixture of the invention, and a method of manufacturing an iron-based sintered body (sintered part) are described.

[0075] The iron-based powder mixture of the invention can be made into a compacted body by a typical compaction method. Specifically, the iron-based powder mixture is filled into a die, and furthermore subjected to compaction. As a typically preferable condition of compaction, pressing force is preferably adjusted to be 400 to 1000 MPa. Moreover, the die may be heated to 50 to 70°C. Alternatively, the powder mixture for powder metallurgy and the die may be heated to 80 to 130°C.

[0076] The iron-based powder mixture of the invention can be adequately compacted in high density even at normal temperature, and preferably compacted at normal temperature in the light of productivity. However, the iron-based powder mixture or the die may be advantageously heated, and the die may be advantageously coated with a lubricant.

[0077] When the powder mixture is compacted in a heated surround, temperature of the iron-based powder mixture or temperature of the die is preferably adjusted to be less than 100°C. The reason for this is that since the iron-based powder mixture according to the invention is high in compressibility, the powder mixture exhibits excellent compaction performance even at a temperature of less than 100°C, and when the temperature is more than 100°C, there is fear that the powder mixture may be degraded due to oxidation. More preferably, the temperature is 80°C or less.

[0078] Next, the high density, iron-based compacted body obtained in the above way is ejected from the die, then subjected to sintering so as to be formed into a high-density sintered body. A sintering method is not particularly limited, and any of previously known sintering methods can be preferably used. In the sintering, preferably, heating temperature is 1100 to 1600°C, and heating time is 10 to 60 min.

[0079] Sintering is performed in this way, thereby a sintered part having excellent strength and excellent machining performance (particularly, a high-strength sintered part in the case of using alloyed steel powder) is obtained.

[0080] After sintering, a sintered part can be subjected to heat treatment such as carburizing and quenching (gas carburizing heat treatment), bright hardening, induction hardening, and carbonitriding heat treatment, so that strength of the (high strength) sintered part can be further increased. Furthermore, tempering may be performed.

50 [Examples]

[0081] Hereinafter, the invention is specifically described according to examples.

[0082] Table 1 shows various types of iron powder for powder metallurgy (each having an average particle diameter of about 80 μ m) used as the iron-based powder in examples 1 to 4. Particularly, in the case of alloyed steel powder, whether the alloyed steel powder is prealloyed steel powder, partly alloyed steel powder, or hybrid steel powder in which the prealloyed steel powder is partly diffused with an alloyed element is distinctively shown.

Table 1

Symbol	Type of iron-based powder	Category of alloyed steel powder
Α	Atomized pure iron powder	-
В	Reduced pure iron powder	•
С	Fe-2%Cu	Partly alloyed steel powder
D	Fe-4%Ni-1.5%Cu-0.5%Mo	Partly alloyed steel powder
E	Fe-2%Ni-1%Mo	Partly alloyed steel powder
F	Fe-0.5%Ni-0.5%Mo	Prealloyed steel powder
G	Fe-0.6%Mo	Prealloyed steel powder
Н	(Fe-0.6%Mol-[0.2%Mo]	Hybrid steel powder*
1	Fe-0.45%Mo	Prealloyed steel powder
J	(Fe-0.45%Mo)-[0.15%Mo]	Hybrid steel powder*
K	(Fe-1.5%Mo)-[2%Ni]	Hybrid steel powder*

^{*} inside of parenthesis: composition of prealloyed steel powder inside of bracket: composition being diffusion-bonded to the prealloyed steel powder

(Example 1)

5

10

15

20

25

30

35

40

45

50

[0083] Various types of iron-based powder as shown in Table 2, and natural graphite powder (average particle diameter of $5~\mu m$) and/or copper powder (average particle diameter of $25~\mu m$) were added with various types of lubricant powder (primary additives), then heated to $140^{\circ} C$ while being mixed by a high-speed mixer, and then cooled to $60^{\circ} C$ or lower, and further added with various types of lubricant powder (secondary additives), and agitated for 1 min at 500~rpm. Then, a powder mixture was discharged from the mixer. A type and blending amount of each of the primary and secondary additives are collectively shown in Table 2. The added amount (part by mass) of a lubricant is expressed in percentage of total mass of 100% of the iron-base powder, natural graphite powder, and copper powder. While the percentage is expressed using a numerical value being not included in that of the total mass, the percentage is approximately the same as in the case that it is expressed using a numerical value being included in that of the total mass. Average particle diameter of the talc powder and average particle diameter of the steatite powder were $6~\mu m$ and $4~\mu m$ respectively.

[0084] For comparison, powder mixtures were prepared (refer to Table 3) in a way that various types of powder having the same components as the above, each including the iron-based powder, and natural graphite powder and/or copper powder, were added with zinc stearate of 0.8 mass%, then the powder was mixed by a V-container-turning mixer. Each of the comparative materials has a composition typically used in normal compaction.

[0085] Next, each of the obtained iron-based powder mixtures was filled in a superhard tablet-shaped die having an inner diameter of 11 mm, and compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of obtained compacted bodies was measured.

[0086] Separately from this, the obtained iron-based powder mixtures were subj ected to compaction for preparing test pieces for a machining test (outer diameter of 60 mm, inner diameter of 20 mm, and length of 30 mm). In the compaction, pressing force was 590 MPa. Sintering was performed in an RX gas atmosphere, wherein heating temperature was 1130°C, and heating time was 20 min. In evaluation of machining performance, while a cermet cutting tool was used, a machining test was performed with cutting speed of 200 m/min, feed of 0.1 mm per unit, depth of cut of 0.3 mm, and a cutting distance of 1000 m, and flank wear width of the cutting tool was measured. Smaller flank wear width of the cutting tool shows more excellent machining performance of a sintered body.

[0087] Obtained results are shown in Table 4.

 55
 45
 40
 35
 30
 25
 20
 15
 10

Table 2

	Iron-ba	ased powder	Natural	Copper powder		Primary ac	ditive lubricant*	Secondary a	dditive lubricant*			
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)			
Inventive	С	99.4	0.6	-	0	STAM	0.1	Steatite	0.1			
example 1						EBS	0.1	STZN	0.02			
						-	-	EBS	0.08			
						-	-	STLI	0.1			
Inventive	А	97.2	0.8	Electrolytic	2.0	STAM	0.1	STZN	0.1			
example 2				copper powder	copper powder		EBS	0.1	EBS	0.02		
						Steatite	0.1	STLI	0.08			
Inventive	Α	97.9	0.6	Atomized	1.5	STAM	0.1	STZN	0.02			
example 3				copper powder		EBS	0.1	EBS	0.08			
						Steatite	0.1	STLI	0.1			
Inventive	В	97.2	0.8	Electrolytic	2.0	STAM	0.1	Steatite	0.1			
example 4				copper powder		EBS	0.1	STZN	0.02			
									-	-	EBS	0.08
						-	-	STLI	0.1			
Inventive	D	99.7	0.3	-	0	STAM	0.1	STZN	0.02			
example 5						EBS	0.1	EBS	0.08			
						Steatite	0.1	STLI	0.1			
Inventive	F	99.5	0.5	-	0	STAM	0.1	Steatite	0.1			
example 6						EBS	0.1	STZN	0.02			
							-	-	EBS	0.08		
						-	-	STLI	0.1			

(continued)

	Iron-ba	sed powder	Natural	Copper powder		Primary a	dditive lubricant*	Secondary	additive lubricant*
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)
Invective	G	97.5	0.5	Electrolytic	=	STAM	0.1	Talc	0.1
example 7				copper powder		EBS	0.1	STZN	0.02
						-	-	EBS	0.08
						-	-	STLI	0.1
Inventive	Н	99.5	0.5	-	0	STAM	0.1	Talc	0.1
example 8						EBS	0.1	STZN	0.02
						-	-	EBS	0.08
						-	-	STLI	0.1
Inventive	I	97.2	0.8	Atomized	2.0	STAM	0.1	Steatite	0.1
example 9				copper powder		EBS	0.1	STAM	0.04
						-	-	EBS	0.04
						-	-	STZN	0.02
						-	-	STLI	0.1

EP 1 985 393 A1

^{*} EBS: ethylene-bis-stearoamide, STZN: zinc stearate, STAM: stearic acid monoamide, STLI: lithium stearate

Table 3

				Table	•		
		Iron-ba	sed powder	Natural	Copper powder		Added
5		type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	lubricant*
Compa exampl		С	99.4	0.6	-	0	0.8 mass% STZN
Compa exampl		А	97.2	0.8	Electrolytic copper powder	2.0	0.8 mass% STZN
Compa exampl		А	97.9	0.6	Atomized copper powder	1.5	0.8 mass% STZN
Compa exampl		В	97.2	0.8	Electrolytic copper powder	2.0	0.8 mass% STZN
Compa exampl		D	99.7	0.3	-	0	0.8 mass% STZN
Compa exampl		F	99.5	0.5	-	0	0.8 mass% STZN
Compa exampl		G	97.5	0.5	Electrolytic copper powder	2.0	0.8 mass% STZN
Compa exampl		Н	99.5	0.5	-	0	0.8 mass% STZN
Compa exampl		1	97.2	0.8	Atomized copper powder	2.0	0.8 mass% STZN
* STZN	: zinc ste	earate		•	1		

Table 4

				l able 4			
35		Iron-based powder	Green density	(Mg/m ³)	Ejection force	(MPa)	Cutting tool
		Туре	490 MPa compaction	686 MPa compaction	490 MPa compaction	686 MPa compaction	Flank wear width (mm)
40	Inventive example 1	С	6.99	7.25	13	18	0.15
	Inventive example 2	А	7.01	7.26	13	18	0.20
45	Inventive example 3	А	7.00	7.26	13	17	0.18
	Inventive example 4	В	6.86	7.09	11	20	0.21
50	Inventive example 5	D	7.00	7.25	13	17	0.48
	Inventive example 6	F	6.91	7.19	14	18	0.15
55	Inventive example 7	G	6.96	7.21	12	18	0.05

(continued)

		Iron-based powder	Green density	(Mg/m ³)	Ejection force	(MPa)	Cutting tool
5		Type	490 MPa compaction	686 MPa compaction	490 MPa compaction	686 MPa compaction	Flank wear width (mm)
	Inventive example 8	Н	6.98	7.22	11	18	0.12
10	Inventive example 9	I	6.98	7.22	12	20	0.03
	Comparative example 1	С	6.96	7.11	11	19	0.35
15	Comparative example 2	A	6.97	7.12	12	18	0.55
	Comparative example 3	A	6.97	7.12	12	17	0.48
20	Comparative example 4	В	6.81	6.99	10	18	0.58
	Comparative example 5	D	6.94	7.15	13	18	0.98
25	Comparative example 6	F	6.84	7.09	14	19	0.43
	Comparative example 7	G	6.85	7.08	13	19	0.22
30	Comparative example 8	Н	6.85	7.09	12	19	0.31
	Comparative example 9	I	6.85	7.09	13	20	0.11

[0088] As clear from a comparison between the inventive examples 1 to 9 and the comparative examples 1 to 9 as shown in Tables 2 to 4, the lubricants according to the invention are used as lubricants, thereby a high-density compacted body can be obtained without significantly increasing ejection force even in the case of normal temperature compaction, and furthermore, machining performance is remarkably improved.

40 (Example 2)

45

50

55

[0089] Various types of iron-based powder as shown in Table 5, and natural graphite powder and/or copper powder were added with various lubricants (primary additives), then heated to 140°C while being mixed by a high-speed mixer, and then cooled to 60°C or lower, and further added with various lubricants (secondary additives), and agitated for 1 min at 500 rpm. Then, a powder mixture was discharged from the mixer. A type and blending amount of each of the primary and secondary additives are collectively shown in Table 5. Used materials are those described in Table 1 as in the example 1.

[0090] For comparison, powder mixtures were prepared in a way that various types of powder having the same components as the above, each including the iron-based powder, and natural graphite powder and/or copper powder, were added with ethylene-bis-stearoamide of 0.6 mass%, then the powder was mixed by a V-container-turning mixer (comparative materials).

[0091] Next, each of the obtained iron-based powder mixtures was filled in a superhard tablet-shaped die having an inner diameter of 11 mm, which was heated beforehand such that temperature of a cavity wall surface was increased to 80°C, and then the powder mixture was compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of the obtained compacted bodies was measured.

[0092] Moreover, each of the comparative materials was compacted at a typical compaction condition of warm com-

paction, that is, the comparative material was heated to 120°C, then filled into a superhard tablet-shaped die having an inner diameter of 11 mm and which was heated to 130°C, and then compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of the obtained compacted bodies was measured.

[0093] Moreover, test pieces for a machining test were prepared by compaction as in the example 1, so that machining performance was examined.

[0094] Obtained results are shown in Table 6.

 55
 45
 40
 35
 30
 25
 20
 15
 10

Table 5

			1	-	ible 5	1		1				
	Iron-ba	ased powder	Natural	Copper powder		Primary add	litive lubricant*	Secondary ad	Iditive lubricant*			
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)			
Inventive	Α	97.2	0.8	Electrolytic	2.0	STAM	0.1	Talc	0.1			
example 10				copper powder		EBS	0.1	STZN	0.02			
						-	-	EBS	0.08			
						-	-	STLI	0.1			
Inventive	D	99.7	0.3	-	0	STAM	0.1	STZN	0.02			
example 11						EBS	0.1	EBS	0.08			
						Steatite	0.1	STLI	0.1			
Inventive	Н	99.35	0.65	-	0	STAM	0.1	Talc	0.1			
example 12						EBS	0.1	STZN	0.02			
						-	-	EBS	0.08			
						-	-	STLI	0.1			
Inventive	E	99.4	0.6	-	0	STAM	0.05	Steatite	0.1			
example 13						I			EBS	0.05	STZN	0.02
						-	-	EBS	0.08			
						-	-	STLI	0.1			
Inventive	J	99.35	0.65	-	0	STAM	0.1	Steatite	0.1			
example 14						EBS	0.1	STAM	0.04			
								-	-	EBS	0.04	
												-
						-	-	STLI	0.1			

 55
 50
 45
 40
 35
 30
 25
 20
 15
 10
 5

(continued)

	Iron-ba	ased powder	Natural	Copper powder		Primary a	dditive lubricant*	Secondary	additive lubricant*	
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	
Inventive	К	99.7	0.3	-	0	STAM	0.05	Steatite	0.1	
example 15						EBS	0.05	STAM	0.04	
						-	-	EBS	0.04	
						-	-	STZN	0.02	
						-	-	STLI	0.1	
Comparative example 10	А	97.2	0.8	Electrolytic copper powder	2.0	0.6 mass	% % EBS	BS		
Comparative example 11	D	99.7	0.3	-	0	0.6 mass	% % EBS			
Comparative example 12	Н	99.35	0.65	-	0	0.6 mass	% % EBS			
Comparative example 13	E	99.4	0.6	-	0	0.6 mass	% % EBS			
Comparative example 14	J	99.35	0.65	-	0	0.6 mass	% % EBS			
Comparative example 15	K	99.7	0.3	-	0	0.6 mass	s% % EBS			

^{*} EBS: ethylene-bis-stearoamide, STZN: zinc stearate, STAM: stearic acid monoamide, STLI: lithium stearate

Table 6

5		Iron-based powder	Green density (M	lg/m³)	Ejection force (MP	a)
J		Туре	490 MPa compaction	686 MPa compaction	490 MPa compaction	686 MPa compaction
10	Inventive example 10	А	7.10	7.30	14	15
10	Inventive example 11	D	7.05	7.30	16	17
	Inventive example 12	Н	7.01	7.26	18	19
15	Inventive example 13	Е	7.04	7.29	15	18
	Inventive example 14	J	7.02	7.27	18	20
20	Inventive example 15	К	6.99	7.24	18	21
	Comparative example 10	А	7.11	7.30	15	16
25	Comparative example 11	D	7.11	7.32	16	18
	Comparative example 12	Н	7.03	7.27	17	20
30	Comparative example 13	Е	7.09	7.30	15	19
	Comparative example 14	J	7.02	7.27	17	19
35	Comparative example 15	К	6.98	7.23	19	22

[0095] As clear from a comparison between the inventive examples 10 to 15 and the comparative examples 10 to 15 as shown in Tables 5 to 6, the primary and secondary additives of the invention were added as lubricants, thereby the die was simply heated to a relatively low temperature of less than 100°C, so that even if the powder mixture was not heated, a compacted body having high density, which was similar to that of a typical warm compacted body, was able to be obtained without causing increase in ejection force.

[0096] Flank wear width (mm) of each inventive example was reduced to about 20 to 40% of that of a comparative example in the same grouping (number), showing remarkable improvement even in machining performance.

(Example 3)

45

50

55

[0097] Various types of iron-based powder as shown in Table 7, and natural graphite powder and/or copper powder were added with various lubricants (primary additives), then heated to 140°C while being mixed by a high-speed mixer, and then cooled to 60°C or lower, and further added with various lubricants (secondary additives), and agitated for 1 min at 500 rpm. Then, a powder mixture was discharged from the mixer. A type and blending amount of each of the primary and secondary additives are collectively shown in Table 7. Used materials are the same as in the example 1. [0098] For comparison, powder mixtures were prepared in a way that each of various types of powder was added with ethylene-bis-stearoamide having a respective weight, then mixed by a V-container-turning mixer.

[0099] Next, each of the obtained iron-based powder mixtures was heated to 60°C, then filled in a superhard tablet-shaped die having an inner diameter of 11 mm, which was heated beforehand such that temperature of a cavity wall surface was increased to 80°C, and furthermore coated with lithium stearate powder on its wall surface, and then the

powder mixture was compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of the obtained compacted bodies was measured. **[0100]** Moreover, each of the comparative materials was compacted at a typical compaction condition of warm compaction, that is, the comparative material was heated to 120°C, then filled into a superhard tablet-shaped die having an inner diameter of 11 mm and which was heated to 130°C, and then compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of the obtained compacted bodies was measured.

[0101] Moreover, test pieces for a machining test were prepared by compaction as in the example 1, so that machining performance was examined.

[0102] Obtained results are shown in Table 8.

55

45

EP 1 985 393 A1

10

15

30 25

	Iron-ba	ased powder	Natural	Copper powder		Primary a	dditive lubricant*	Secondary	additive lubricant*	
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	
Inventive	Α	97.2	0.8	Electrolytic	2.0	STAM	0.2	Steatite	0.2	
example 16				copper powder		EBS	0.2	STZN	0.04	
						-	-	EBS	0.16	
Inventive	G	99.35	0.65	-	0	STAM	0.1	Talc	0.1	
example 17						EBS	0.1	STZN	0.02	
						-	-	EBS	0.08	
						-		STLI	0.1	
Comparative example 16	A	97.2	0.8	Electrolytic copper powder	2.0	0.8 mass	% EBS	1		
Comparative example 17	G	99.35	0.65	-	0	0.6 mass	ass % EBS			

^{*} EBS: ethylene-bis-stearoamide, STZN: zinc stearate, STAM: stearic acid monoamide, STLI: lithium stearate

Table 8

5		Iron- based powder	Compaction temperature		Green density	(Mg/m³)	Ejection force	(MPa)
		Туре	Powder (°C)	Die (°C)	490 MPa compaction	686 MPa compaction	490 MPa compaction	686 MPa compaction
10	Inventive example 16	A	60	80	6.90	7.19	8	11
	Inventive example 17	G	60	80	7.04	7.29	15	19
15	Comparative example 16	A	120	130	6.94	7.2	9	12
	Comparative example 17	G	120	130	7.00	7.26	16	19

[0103] As clear from a comparison between the inventive example 16 and the comparative example 16, and a comparison between the inventive example 17 and the comparative example 17 as shown in Tables 7 to 8, the primary and secondary additives of the invention were added as lubricants, thereby the die and the powder were simply heated to a relatively low temperature of less than 100°C, so that a compacted body having high density, which was similar to that of a typical warm compacted body, was able to be obtained with an extremely low ejection force.

[0104] Flank wear width (mm) of each inventive example was reduced to about 25 to 35% of that of a comparative example in the same grouping (number), showing remarkable improvement even in machining performance.

(Example 4)

[0105] Various types of iron-based powder as shown in Table 9, and natural graphite powder and/or copper powder were added with various lubricants (primary additives), then heated to 140°C while being mixed by a high-speed mixer, and then cooled to 60°C or lower, and further added with various lubricants (secondary additives), and agitated for 1 min at 500 rpm. Then, a powder mixture was discharged from the mixer. A type and blending amount of each of the primary and secondary additives are collectively shown in Table 9. Used materials are the same as in the example 1. A comparative example 20 was subjected to processing where the relevant powder was added with steatite powder in place of the primary and secondary additives, then mixed by the high-speed mixer at the same condition as in the above. [0106] Next, each of the obtained iron-based powder mixtures was filled in a superhard tablet-shaped die having an inner diameter of 11 mm, and compacted at 490 MPa and 686 MPa. In such compaction, when a compacted body was ejected from the die, ejection force was measured, and green density of each of obtained compacted bodies was measured.

[0107] Separately from this, the obtained iron-based powder mixtures were subjected to compaction for preparing tensile test pieces according to Japan Powder Metallurgy Association JPMA M04-1992, and test pieces for a machining test (outer diameter of 60 mm, inner diameter of 20 mm, and length of 30 mm). In the compaction, pressing force was 590 MPa. Sintering was performed in an RX gas atmosphere, wherein heating temperature was 1130°C, and heating time was 20 min. An evaluation method of machining performance was the same as in the example 1.

[0108] Obtained results are shown in Table 10.

50

45

 55
 50
 45
 40
 35
 30
 25
 20
 15
 10
 5

Table 9

	Iron-base	ed powder graphite	Natural	Copper powde	er	Prima add	ditive lubricant*	Secondary a	additive lubricant*
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)
Comparative	D	99.4	0.6	-	0	STAM	0.1	STLI	0.1
example 18						EBS	0.1	STZ	0.02
						-	-	EBS	0.08
Inventive	D	99.4	0.6	-	0	STAM	0.1	Steatite	0.05
example 18						EBS	0.1	STLI	0.1
						-	-	STZN	0.02
							-	EBS	0.08
Inventive	D	99.4	0.6	-	0	STAM	0.1	Steatite	0.1
example 19						EBS	0.1	STLI	0.1
						-	-	STZN	0.02
							-	EBS	0.08
Inventive	D	99.4	0.6	-	0	STAM	0.1	Steatite	0.2
example 20						EBS	0.1	STLI	0.1
						-	-	STZN	0.02
						-	-	EBS	0.08
Inventive	D	99.4	0.6	-	0	STAM	0.1	Steatite	0.3
example 21						EBS	0.1	STLI	0.1
						-	-	STZN	0.02
						-	-	EBS	0.08

(continued)

	Iron-base	ed powder graphite	l · · · ·		Prima add	itive lubricant*	Secondary a	Secondary additive lubricant*	
	type	Blending amount (mass%)	graphite powder (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)	type	Blending amount (mass%)
Comparative	D	99.4	0.6	-	0	STAM	0.1	Steatite	0.6
example 19						EBS	0.1	STLI	0.1
						-	-	STZN	0.02
				-	-	EBS	0.08		
Inventive example 22	1	97.2	0.8	Electrolytic copper powder	2.0	STAM	0.1	Steatite	0.1
						EBS	0.1	STLI	0.1
						STZN	0.1	-	-
Comparative example 20	I	97.2	0.8	Electrolytic copper powder	2.0	Steatite	0.1	-	-
Inventive	I	97.2	0.8	Electrolytic	2.0	STAM	0.2	Steatite	0.1
example 23	23 copper powder	copper powder		EBS	0.2	-	-		
Comparative	ı	97.2	0.8	Electrolytic copper powder	2.0	STZN	0.2	Steatite	0.1
example 21								STLI	0.2

EP 1 985 393 A1

^{*} EBS: ethylene-bis-stearoamide, STZN: zinc stearate, STAM: stearic acid monoamide, STLI: lithium stearate

Table 10

5		Iron- based powder	Green density	/ (Mg/m ³)	Ejection force (MPa)		Sintered body	Cutting tool	
		Туре	490 MPa compaction	686 MPa compaction	490 MPa compaction	686 MPa compaction	Tensile strength (MPa)	Flank wear width (mm)	
10	Comparative example 18	D	6.99	7.24	14	18	630	0.92	
	Inventive example 18	D	6.99	7.25	13	17	625	0.65	
15	Inventive example 19	D	7.00	7.25	12	16	610	0.54	
	Inventive example 20	D	6.97	7.23	13	17	590	0.51	
20	Inventive example 21	D	6.95	7.21	13	18	550	0.45	
	Comparative example 19	D	6.94	7.20	15	19	490	0.43	
25	Inventive example 22	I	6.99	7.23	12	18	620	0.03	
	Comparative example 20	I	Compaction is disabled due to galling.						
30	Inventive example 23	1	6.98	7.22	13	19	630	0.05	
	Comparative example 21	I	6.96	7.20	20	20	610	0.06	

[0109] As clear from a comparison between the inventive examples 18 to 21 and the comparative examples 18 and 19 as shown in Tables 9 to 10, in the iron-based powder mixture in which steatite and the like are added within a range of the invention, a high-density compacted body can be obtained without increasing ejection force. In the comparative example 19 in which steatite and the like are added in a range of more than 0.5 mass%, mechanical properties are significantly reduced. Furthermore, it is known from the inventive examples 18 to 21 that the added amount of steatite and the like is more preferably 0.2 mass% or less in the light of the mechanical properties.

[0110] As clear from a comparison between the inventive examples 22 and 23 and the comparative examples 20 and 21, fatty acid amide and the like need to be added with steatite and the like to obtain a high-density compacted body without increasing ejection force. Moreover, it is known that metallic soap is further added, thereby machining performance of a sintered body can be remarkably improved.

(Example 5)

35

45

50

55

[0111] Water-atomized alloyed steel powder having a composition shown in Table 11 was manufactured by a water-atomizing method. The rest of the powder other than Mn and Mo is Fe and inevitable impurities. The water-atomized alloyed steel powder was added with Cu powder, graphite powder, talc, and steatite in a ratio as shown in the Table 11. Each of Mo content and Mn content (mass%) in the water-atomized steel powder, or each of the added amount (mass%) of the Cu powder, graphite powder, talc, and steatite to be added to the water-atomized steel powder is shown in percentage of mass of a powder mixture for powder metallurgy, the percentage being expressed using a numerical value included in a numerical value of mass of the powder mixture.

[0112] Furthermore, a lubricant was added in a ratio as shown in Table 11. The added amount (part by mass) of the lubricant is shown in percentage of mass (100 part by mass) of a powder mixture for powder metallurgy obtained by mixing the water-atomized alloyed steel powder and additives, the percentage being expressed using a numerical value

being not included in a numerical value of moss of the powder mixture (but, the percentage is approximately the same as in the case that it is expressed using a numerical value included therein).

[0113] Next, the materials were mixed by a V blender, then an obtained powder mixture for powder metallurgy was filled in a die so as to be subjected to compaction for preparing tensile test pieces according to Japan Powder Metallurgy Association JPMA M04-1992, and test pieces for a machining test (outer diameter of 60 mm, inner diameter of 20 mm, and length of 30 mm). In the compaction, pressing force was 590 MPa. Sintering was performed in an RX gas atmosphere, wherein heating temperature was 1130°C, and heating time was 20 min.

[0114] Tensile strength obtained by a tensile test was as shown in Table 11.

[0115] In evaluation of machining performance, while a cermet cutting tool was used, a machining test was performed with cutting speed of 200 m/min, feed of 0.1 mm per unit, depth of cut of 0.3 mm, and a cutting distance of 1000 m, and flank wear width of the cutting tool was measured. Results of the measurement are as shown in Table 11. Smaller flank wear width of the cutting tool shows more excellent machining performance of a sintered body.

[0116] In Table 11, inventive examples use a powder mixture for powder metallurgy that satisfies the scope of the invention, and comparative examples use a powder mixture for powder metallurgy that departs from the scope of the invention. In a prior-art example of No. 22, a powder mixture for powder metallurgy using Fe-4Ni-1.5Cu-0.5Mo water-atomized alloyed steel powder, which is previously practically used, is blended with a conventional lubricant. Numerical values added to respective alloy elements of No. 22 are expressed in mass%.

Table 11

No	Powder m	nixture for pov	vder metallur	gy (mass%)*1			Lubricant	:1	Lubrican	t 2	Sintered Cut	ting body tool	Remarks
	Water-ato alloyed st	mized eel powder Mn	Cu powder	Graphite powder	Talc	Steatite	Type *2	Added amount (part by mass) *3	Type *2	Added amount (part by mass) *3	Tensile strength (MPa)	Flank wear width (mm)	
1	0.45	0.21	0.0	0.8	0.1	-	EBS	0.4	-	-	380	0.04	Inventive example
2	0.45	0.21	1.5	0.8	0.1	-	EBS	0.4	-	-	520	0.08	Inventive example
3	0.45	0.21	2.0	0.8	0.1	-	EBS	0.4	-	-	630	0.08	Inventive example
4	0.45	0.21	3.0	0.8	0.1	-	EBS	0.4	-	-	650	0.15	Inventive example
6	0.45	0.05	2.0	0.8	-	0.1	STAM	0.2	STZN	0.2	480	0.02	Comparative example
7	0.45	0.12	2.0	0.8	-	0.1	STAM	0.2	STZN	0.2	550	0.02	Inventive example
8	0.45	0.19	2.0	0.8	-	0.1	STAM	0.2	STZN	0.2	640	0.04	Inventive examplee
10	0.2	0.20	2.0	0.8	0.1	0.1	STAM	0.4	-	-	430	0.07	Inventive example
11	0.3	0.20	2.0	0.8	0.1	0.1	STAM	0.4	-	-	540	0.07	Inventive example
12	0.5	0.20	2.0	0.8	0.1	0.1	STAM	0.4	-	-	630	0.07	Inventive example
14	0.45	0.21	2.0	0.4	0.1	-	EBS	0.5	-	-	410	0.05	Inventive example
15	0.45	0.21	2.0	0.6	0.1	-	EBS	0.5	-	-	530	0.05	Inventive example
16	0.45	0.21	2.0	0.9	0.1	-	EBS	0.5	-	-	620	0.10	Inventive example

(continued)

No	Powder mixture for powder metallurgy (mass%)*1				Lubricant	Lubricant 1 Lu		Lubricant 2		tting body tool	Remarks		
		teel powder	Cu powder	Graphite powder	Talc	Steatite	Type *2	Added amount (part by	Type *2	Added amount (part by	Tensile strength (MPa)	Flank wear width (mm)	
	Мо	Mn						mass) *3		mass) *3	(wii a)		
18	0.45	0.21	2.0	0.8	-	-	EBS	0.3	STLI	0.15	650	0.30	Comparative example
19	0.45	0.21	2.0	0.8	-	0.3	EBS	0.3	STLI	0.15	640	0.02	Inventive example
20	0.45	0.21	2.0	0.8	-	0.5	EBS	0.3	STLI	0.15	600	0.02	Inventive example
21	0.45	0.21	2.0	0.8	-	0.7	EBS	0.3	STLI	0.15	460	0.01	Comparative example
22	Fe-4Ni-1.	5Cu-05Mo		0.6	-		EBS	0.5	EBS	0.5	590	0.48	Prior-art example

^{*1} percentage of mass of powder mixture for powder metallurgy (by a numerical value included in a numerical value of the mass)

^{*2} EBS: ethylene-bis-stearoamide, STZN: zinc stearate, STAM: stearic acid monoamide, STLI: lithium stearate

^{*3} percentage of mass of powder mixture for powder metallurgy (by a numerical value not included in a numerical value of the mass)

[0117] As obvious from Table 11, particularly, any of sintered bodies obtained from the powder mixtures for powder metallurgy of the inventive examples are excellent in mechanical properties and machining performance. The prior-art example is particularly significantly bad in machining performance of a sintered body.

[0118] When the water-atomized alloyed steel powder contains Mo of 0.3 to 0.5 mass% and Mn of 0.1 to 0.25 mass%, and the powder mixture contains Cu powder of 1 to 3 mass% and graphite powder of 0.5 to 1.0 mass%, a sintered body can be obtained, which has a tensile strength of 500 MPa or more, and is excellent in machining performance.

Industrial Applicability

- 10 [0119] According to the invention, an iron-based powder mixture can be obtained, which gives high compaction density and small ejection force even if the powder mixture is compacted at low temperature of about room temperature. Moreover, according to a preferred invention, a powder mixture for powder metallurgy can be obtained, which is preferable for machining a sintered part having excellent machining performance, particularly, preferable for machining a high-strength sintered part.
- [0120] Moreover, according to the invention, the iron-based powder mixture is used as a material, thereby an iron-based compacted body having high compaction density can be obtained, and furthermore, an iron-based sintered body can be obtained, which has high sintering density, or has further excellent machining performance.

20 Claims

25

35

40

45

50

55

1. An iron-based powder mixture containing:

an iron-based powder, and containing as additives,

- at least one selected from talc and steatite, and
- fatty acid amide.
- **2.** The iron-based powder mixture according to claim 1:

wherein the additives further contain metallic soap.

3. The iron-based powder mixture according to claim 1:

wherein the iron-based powder mixture is further blended with alloying powder.

4. The iron-based powder mixture according to claim 2:

wherein the iron-based powder mixture is further blended with alloying powder.

5. The iron-based powder mixture according to claim 3 or 4:

wherein the iron-based powder is water-atomized alloyed steel powder containing Mo of 0.3 to 0.5 mass%, Mn of 0.1 to 0.25 mass%, and the remainder being Fe and inevitable impurities, and wherein the alloying powder is Cu powder of 1 to 3 mass% and graphite powder of 0.5 to 1.0 mass%.

6. An iron-based powder mixture, characterized in that:

the iron-based powder mixture is formed by mixing water-atomized alloyed steel powder containing Mo of 0. 3 to 0.5 mass%, Mn of 0.1 to 0.25 mass%, and the remainder being Fe and inevitable impurities,

Cu powder of 1 to 3 mass%,

graphite powder of 0.5 to 1.0 mass%,

at least one selected from talc and steatite in a range of 0.05 to 0.5 mass% in total, and fatty acid amide.

7. The iron-based powder mixture according to claim 6:

wherein the iron-based powder mixture further contains metallic soap.

	8.	A method of manufacturing an iron-based compacted body, characterized in that :
5		the iron-based powder mixture according to any one of claims 1 to 4 is filled in a die, then compacted at a temperature of less than 100°C.
	9.	A method of manufacturing an iron-based sintered body, characterized in that:
10		the iron-based powder mixture according to any one of claims 1 to 4 is filled in a die, then compacted at a temperature of less than 100°C, and then an obtained iron-based compacted body is sintered.
15		
20		
25		
30		
35		
40		
45		
50		
55		

INTERNATIONAL SEARCH REPORT

International application No.

		PCT/JP:	2007/053125
	TATION OF SUBJECT MATTER 2006.01)i, B22F1/00(2006.01)i, n	C22C33/02(2006.01)n,	C22C38/00
According to Inte	ernational Patent Classification (IPC) or to both national	al classification and IPC	
B. FIELDS SE	ARCHED		
Minimum docun B22F1/00-	nentation searched (classification system followed by cl 8/00, C22C33/02, C22C38/00	assification symbols)	
Jitsuyo		ent that such documents are included in tsuyo Shinan Toroku Koho roku Jitsuyo Shinan Koho	the fields searched 1996-2007 1994-2007
Electronic data b	pase consulted during the international search (name of	data base and, where practicable, search	n terms used)
C. DOCUMEN	ITS CONSIDERED TO BE RELEVANT		1
Category*	Citation of document, with indication, where ap		Relevant to claim No.
X A	JP 2005-264201 A (JFE Steel 29 September, 2005 (29.09.05) Claim 1; Par. Nos. [0003], [0 [0027] & WO 2005/087411 A1) ,	1-4,8,9 5,6,7
A	JP 2005-15866 A (Mitsubishi 20 January, 2005 (20.01.05), Claim 7 & WO 2005/001150 A1	Materials Corp.),	5,6,7
	ocuments are listed in the continuation of Box C.	See patent family annex.	
"A" document de be of particu "E" earlier applie date "L" document we cited to esta special reaso "O" document re: "P" document pupriority date Date of the actual 22 June	cation or patent but published on or after the international filing which may throw doubts on priority claim(s) or which is blish the publication date of another citation or other in (as specified) ferring to an oral disclosure, use, exhibition or other means abbished prior to the international filing date but later than the claimed all completion of the international search all completion (22.06.07)	"T" later document published after the inte date and not in conflict with the applicate the principle or theory underlying the in document of particular relevance; the considered novel or cannot be consisted when the document is taken alone "Y" document of particular relevance; the considered to involve an inventive stombined with one or more other such being obvious to a person skilled in the "&" document member of the same patent for Date of mailing of the international se 03 July, 2007 (03.	tion but cited to understand vention laimed invention cannot be lered to involve an inventive laimed invention cannot be ep when the document is documents, such combination art amily
	ng address of the ISA/ se Patent Office	Authorized officer Telephone No.	
L'acommic INO.		1 stephone 1 to.	

Facsimile No.
Form PCT/ISA/210 (second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No. PCT/JP2007/053125

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)							
This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons: 1. Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:							
Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:							
3. Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).							
Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)							
This International Searching Authority found multiple inventions in this international application, as follows: Claim 1 relates to an iron-base mixed powder comprising an iron-base powder at least one of talc and steatite, and a fatty acid amide. Claims 2 and 4 relate to an iron-base mixed powder as set forth in claim 1 which contains a metal soap. Claim 3 relates to an iron-base mixed powder as set forth in claim 1 which contains an alloying powder. Claims 5-7 relate to an iron-base mixed powder as set forth in claim 1 which is specified in composition. Claim 8 relates to a process for the production of iron-base powder compacts (continued to extra sheet) 1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.							
any additional fee. 3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:							
4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:							
Remark on Protest The additional search fees were accompanied by the applicant's protest and, where applicable,							
the payment of a protest fee. The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation. No protest accompanied the payment of additional search fees.							

Form PCT/ISA/210 (continuation of first sheet (2)) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2007/053125

Claim 9 relates to a process for the production of sintered iron-base powder compacts.	Continuation of Box No.III of continuation of first sheet(2)
powder compacts.	Claim 0 moletageto a process for the production of giptored iron bago
	powder compacts
	powder compaces.

Form PCT/ISA/210 (extra sheet) (April 2005)

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- US 5256185 A [0006]
- JP 2156002 A [0009] [0010]
- JP 7103404 B **[0009] [0010]**
- US 5368630 A [0009]
- US P5368630 A [0010]
- JP 9104901 A **[0011]**

- JP 10317001 A [0011]
- JP 459649 B [0015] [0018]
- JP 61163239 A [0015] [0018]
- JP 63114903 A [0015] [0018]
- JP 1255604 A **[0019] [0020]**
- JP 6479302 A [0019] [0020]