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(54) PROCESS FOR PRODUCTION OF SINTERED COMPACT BY POWDER METALLURGY

(57) In a method for producing a sintered compact by powder metallurgy, a high-strength sintered compact is produced at low cost using an alloy steel powder for powder metallurgy containing no nickel or copper by compacting the alloy steel powder for powder metallurgy or a mixed powder containing the alloy steel powder at a pressure of 700 MPa or more and sintering the compact

at a temperature of 1,150°C to 1,300°C. The alloy steel powder used contains 0.3% to 0.7% by mass of chromium, 0.1% to 0.5% by mass of manganese, 0.1% to 0.5% by mass of molybdenum, and 0.25% to 0.5% by mass of oxygen, the balance being iron and incidental impurities.

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Description

Technical Field

⁵ **[0001]** The present invention relates to methods for producing sintered compacts suitable, for example, for automobile high-strength sintered parts by powder metallurgy.

Background Art

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[0002] Powder metallurgy, in which an iron powder or alloy steel powder is compacted in a die under pressure and is then sintered to form a sintered compact, is widely used for production of automobile parts, such as gears, which require high dimensional accuracy. Sintered compacts for use as automobile parts are produced by charging a mixture of a raw material powder (iron powder or alloy steel powder) with, for example, a copper powder, a graphite powder, and a lubricant into a die, compacting the mixed powder in the die under pressure, and sintering the compact, and typically have a density of about 6.0 to 7.2 g/cm³.

[0003] For production of parts requiring high strength among automobile parts (hereinafter referred to as automobile high-strength sintered parts), techniques in which alloying elements (such as nickel, copper, molybdenum, chromium, and manganese) are added to an iron-based powder for use as a raw material powder have been put to practical use. The procedure of mixing the raw material powder with, for example, a copper powder, a graphite powder, and a lubricant into a die, compacting the mixed powder in the die under pressure, and sintering the compact is similar to the procedure for typical automobile parts as described above. For automobile high-strength sintered parts, however, treatment such as bright quenching and tempering and carburizing is optionally performed.

[0004] Examples of methods for adding alloying elements include alloying an iron-based powder in advance (i.e., prealloying), mixing an iron-based powder with an alloying powder containing desired alloying elements and a binder, mixing an iron-based powder with an alloying powder (without a binder), and mixing an iron-based powder with an alloying powder and maintaining the mixed powder at high temperature so that it bonds metallurgically (i.e., diffusion bonding). Alloy steel powders (or mixed powders) prepared by these methods differ in the properties thereof, the uniformity of the alloying elements, and the state of diffusion of the alloying elements after sintering. Hence, the selection of the alloying elements and the method for adding them is an important factor that greatly affects the quality of a sintered compact.

[0005] For example, Patent Literature 1 discloses a technique using a raw material powder prepared by diffusion bonding of metal powders such as nickel, copper, and molybdenum powders to an iron powder. According to this technique, the raw material powder is sintered after compaction (pressure compaction) so that alloying elements such as nickel, copper, and molybdenum diffuse from the metal powders adhering to the surfaces of the iron powder into the iron powder to alloy the iron powder. However, sufficiently alloying the iron powder requires sintering over an extended period of time because the diffusion of the alloying elements such as nickel, copper, and molybdenum takes an extended period of time, thus decreasing the productivity of sintering. Furthermore, large amounts of metal powders such as nickel, copper, and molybdenum powders must be used to attain the strength required for automobile high-strength sintered parts, thus increasing the raw material costs.

[0006] In addition, a sintered compact produced by the technique disclosed in Patent Literature 1 contains nickel and copper; nickel is harmful to the human body, and copper has a problem in that it accumulates in scraps as a harmful element if the steel is recycled. Accordingly, alloy steel powders containing no nickel or copper have been studied. Patent Literature 2, for example, discloses an alloy steel powder containing 0.2% to 1.4% by mass of molybdenum, 0.1% to 0.3% by mass of chromium, 0.10% by mass or less of carbon, and 0.3% by mass or less of oxygen. A sintered compact produced from the alloy steel powder has relatively high strength because chromium and molybdenum have the effect of improving quench hardenability.

[0007] Patent Literature 3 discloses an alloy steel powder containing 0.1% by mass or less of carbon, 0.08% by mass or less of manganese, 0.5% to 3% by mass of chromium, 0.1% to 2% by mass of molybdenum, 0.01% by mass or less of sulfur, 0.2% by mass or less of phosphorus, and 0.2% by mass or less of oxygen.

[0008] Patent Literature 4 discloses an alloy steel powder containing 1.3% to 1.7% by mass of chromium, 0.15% to 0.3% by mass of molybdenum, 0.09% to 0.3% by mass of manganese, 0.01% by mass or less of carbon, and 0.25% by mass or less of oxygen.

Citation List

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Patent Literature

[0009]

- PTL 1: Japanese Examined Patent Application Publication No. 45-9649
- PTL 2: Japanese Unexamined Patent Application Publication No. 61-117201
- PTL 3: Japanese Unexamined Patent Application Publication No. 5-287452
 - PTL 4: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2005-530037

Summary of Invention

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Problems to be solved by the Invention

[0010] With the alloy steel powders disclosed in Patent Literatures 2 and 3, however, it is not easy to attain the high strength required for automobile high-strength sintered parts. For the alloy steel powder in Patent Literature 2, the above problem can be solved by, for example, increasing the chromium and molybdenum contents, although large amounts of chromium and molybdenum added decrease the compressibility of the alloy steel powder and increase the raw material costs. For the alloy steel powder in Patent Literature 3, the strength problem can be solved by, for example, increasing the manganese content, although a large amount of manganese added results in formation of oxide during sintering after compaction or during heat treatment after sintering because manganese is a readily oxidizable element, thus decreasing the strength of a sintered compact. For the alloy steel powder in Patent Literature 4, its high chromium content decreases the compressibility of the alloy steel powder, thus decreasing the density of a sintered compact. In addition, a large amount of chromium added results in formation of oxide during sintering after compaction or during heat treatment after sintering because chromium is a readily oxidizable element, thus decreasing the strength of a sintered compact. An object of the present invention is to solve the above problems in the conventional art and to provide a method for producing a high-strength sintered compact by powder metallurgy at low cost using an alloy steel powder for powder metallurgy containing no nickel or copper.

Solution to Problem

30 [0011] The inventors have studied in detail the component conditions of alloy steel powders for powder metallurgy and the production conditions of sintered compacts for solving the above problems. As a result, the inventors have found that a high-strength sintered compact that can be used as an automobile high-strength sintered part can be produced at low cost using an alloy steel powder for powder metallurgy containing no nickel or copper and containing a relatively small amount of molybdenum by optimizing the chromium, manganese, and molybdenum contents of an alloy steel powder for powder metallurgy and subjecting the raw material powder to high-pressure compaction and high-temperature sintering under particular conditions.

[0012] It has also been found that even an alloy steel powder having a fairly high oxygen content can be used without a substantial decrease in the strength of a sintered compact by adding a sufficient amount of graphite powder to the alloy steel powder so that graphite reacts with oxygen to reduce the alloy steel powder in sintering. That is, the above alloy steel powder for powder metallurgy, containing chromium and manganese, which are readily oxidizable elements, must be subjected to reduction treatment in a reduced-pressure atmosphere at a high temperature, namely, about 1,100°C, in order to lower the oxygen content thereof. However, it has been found that even an alloy steel powder having a fairly high oxygen content after low-cost reduction treatment in a hydrogen gas atmosphere, rather than the above costly reduction treatment, can be used without a substantial decrease in the strength of a sintered compact by adding a sufficient amount of graphite powder so that graphite reacts with oxygen to reduce the alloy steel powder, from which oxygen is removed, during sintering.

This technique, however, might cause variations in the amount of carbon remaining in the sintered compact. As a result of a further study, it has been found that the carbon content of the sintered compact, which significantly affects the strength of the sintered compact, can be controlled by supplementarily adding the amount of graphite determined from the chromium and manganese contents of the alloy steel powder and the oxidation rates of these elements because the amount of graphite used for reduction of oxygen in the alloy steel powder during sintering is mainly determined by the chromium and manganese contents of the alloy steel powder and the oxidation rates of these elements.

[0013] The present invention is based on these findings and is summarized as follows:

[1] A method for producing a sintered compact by powder metallurgy, including compacting an alloy steel powder for powder metallurgy or a mixed powder containing the alloy steel powder at a pressure of 700 MPa or more and sintering the compact at a temperature of 1,150°C to 1,300°C, wherein the alloy steel powder used contains 0.3% to 0.7% by mass of chromium, 0.1% to 0.5% by mass of manganese, 0.1% to 0.5% by mass of molybdenum, and

- 0.25% to 0.5% by mass of oxygen, the balance being iron and incidental impurities.
- [2] The method for producing a sintered compact according to Item [1] above, wherein the mixed powder contains a graphite powder.
- [3] The method for producing a sintered compact according to Item [1] or [2] above, wherein the mixed powder contains a graphite powder in an amount equivalent to the sum of the amount of carbon allowed to remain in the sintered compact and the amount of carbon that reacts with oxygen in the alloy steel powder for powder metallurgy in the sintering.

[0014]

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[4] The method for producing a sintered compact according to Item [2] above, wherein the graphite powder is added to the alloy steel powder for powder metallurgy in an amount [%Gr] (% by mass based on the mass of the alloy steel powder for powder metallurgy) satisfying the condition of equation (1):

[%Gr] = t × (
$$\alpha$$
 × 0.46 × [%Cr] + β × 0.29 × [%Mn]) + γ + [%C] ... (1)

wherein

[%Cr] is the chromium content of the alloy steel powder for powder metallurgy (% by mass);

[%Mn] is the manganese content of the alloy steel powder for powder metallurgy (% by mass);

[%C] is the amount of carbon, in the graphite powder, allowed to remain in the sintered compact (% by mass based on the mass of the alloy steel powder for powder metallurgy);

 α is the oxidation rate of chromium in the alloy steel powder for powder metallurgy;

β is the oxidation rate of manganese in the alloy steel powder for powder metallurgy;

 γ is the amount of carbon, in the graphite powder, consumed by reaction with oxygen adsorbed on the alloy steel powder for powder metallurgy and oxygen contained in a sintering atmosphere (% by mass based on the mass of the alloy steel powder for powder metallurgy), wherein $\gamma \le 0.2\%$ by mass; and t is 0.25 to 0.75.

Advantageous Effects of Invention

[0015] According to the present invention, a high-strength sintered compact that can be used as an automobile high-strength sintered part can be produced at low cost by a method for producing a sintered compact by powder metallurgy using an alloy steel powder containing no nickel or copper and containing a relatively small amount of molybdenum.

Brief Description of Drawing

[0016] [Fig. 1] Fig. 1 is a graph contrasting the relationships between compaction pressure and tensile strength of Sample Nos. 2 and 5 to 7 in Example 2 with those of Sample Nos. 8 to 11, which are existing high-strength materials.

Description of Embodiments

[0017] First, the chemical components of an alloy steel powder for powder metallurgy used in the present invention (hereinafter also simply referred to as "alloy steel powder") and the reasons for limitations thereon will be described.

Chromium: 0.3% to 0.7% by mass

[0018] Chromium, an element that improves quench hardenability, provides the effect of increasing the strength of a sintered compact by causing martensitic transformation during quenching after sintering. If the chromium content falls below 0.3% by mass, the effect is insufficient. On the other hand, if the chromium content exceeds 0.7% by mass, the compressibility in compaction is decreased because the hardness of the individual particles is increased by solid solution hardening and the oxygen content of the alloy steel powder is increased. In addition, it is impossible to attain a significant improvement in the strength of a sintered compact due to improved quench hardenability because more oxidation occurs in the atmosphere during quenching after sintering; rather, the decreased compressibility significantly decreases the strength of a sintered compact. Accordingly, the chromium content is 0.3% to 0.7% by mass.

Manganese: 0.1% to 0.5% by mass

[0019] Manganese, an element that improves quench hardenability, provides the effect of increasing the strength of a sintered compact by causing martensitic transformation during quenching after sintering. If the manganese content falls below 0.1% by mass, the effect is insufficient. On the other hand, if the manganese content exceeds 0.5% by mass, the compressibility in compaction is decreased because the hardness of the individual particles is increased by solid solution hardening and the oxygen content of the alloy steel powder is increased, thus decreasing the strength of a sintered compact. In addition, for sintering in a weakly oxidizing atmosphere (such as a hydrocarbon-converted gas atmosphere), if the manganese content is excessive, it is impossible to attain a significant improvement in the strength of a sintered compact due to improved quench hardenability because more oxidation occurs in the atmosphere during quenching after sintering; rather, the decreased compressibility significantly decreases the strength of a sintered compact. Accordingly, the manganese content is 0.1% to 0.5% by mass, preferably 0.1% to 0.25% by mass.

Molybdenum: 0.1% to 0.5% by mass

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[0020] Molybdenum provides the effect of increasing the strength of a sintered compact by increasing the quench hardenability and facilitating solid solution hardening and precipitation hardening. If the molybdenum content falls below 0.1% by mass, the effect is insufficient. On the other hand, if the molybdenum content exceeds 0.5% by mass, it is impossible to attain a significant improvement in the strength of a sintered compact because the compressibility in compaction is decreased; rather, the raw material costs are considerably increased with increasing amount of molybdenum added. Accordingly, the molybdenum content is 0.1% to 0.5% by mass.

Oxygen: 0.25% to 0.5% by mass

[0021] Lowering the oxygen content to less than 0.25% by mass requires reduction treatment in a very clean reducing atmosphere or reduced-pressure atmosphere, thus increasing the production costs. On the other hand, if the oxygen content exceeds 0.5% by mass, the strength of a sintered compact is decreased because the compressibility is decreased and sintering is inhibited. Accordingly, the oxygen content is 0.25% to 0.5% by mass, preferably 0.25% to 0.35% by mass. In general, lowering the oxygen content of an alloy steel powder containing readily oxidizable elements such as chromium and manganese requires reduction treatment in a very clean reducing atmosphere or reduced-pressure atmosphere, thus increasing the production costs. In contrast, the inventors have found that oxygen in an alloy steel powder can be reduced in a subsequent sintering step even if the alloy steel powder has a fairly high oxygen content after reduction treatment in a low-cost normal reducing atmosphere. In the present invention, therefore, the oxygen content of the alloy steel powder is 0.25% to 0.5% by mass, which is higher than those of common alloy steel powders containing readily oxidizable elements.

The balance other than the components described above is iron and incidental impurities. The alloy steel powder used in the present invention preferably has a carbon content of 0.01% by mass or less, more preferably 0.005% by mass or less. If the carbon content exceeds 0.01% by mass, the compressibility in compaction tends to be decreased because the hardness of the individual particles of the alloy steel powder is excessively increased, thus decreasing the strength of a sintered compact.

[0022] For example, the above alloy steel powder is produced as follows.

A molten alloy steel containing predetermined chemical components is prepared, and an alloy steel powder is produced by water atomization. In water atomization, no particular limitation is imposed on the operating conditions and the configuration of the equipment used, and a known technique may be used. The alloy steel powder is then subjected to reduction heat treatment. In the reduction heat treatment, no particular limitation is imposed on the operating conditions and the configuration of the equipment used, and a known technique may be used to perform the treatment in a reducing atmosphere such as a hydrogen gas atmosphere or a vacuum atmosphere. In the present invention, the reduction treatment can be performed in a low-cost hydrogen gas atmosphere because a graphite powder optionally added to the alloy steel powder removes oxygen from the alloy steel powder in a subsequent sintering step. The reduction treatment temperature is exemplified by, but not limited to, about 800°C to 1,000°C. In view of cost, 950°C or less is preferred.

[0023] Next, the production conditions of the present invention will be described.

In the present invention, a sintered compact is obtained by optionally mixing the alloy steel powder described above with one or more of, for example, other metal powders, a graphite powder, and a lubricant, charging the alloy steel powder or mixed powder containing the alloy steel powder into a die, compacting the powder in the die under pressure, and sintering the compact.

The lubricant used may be, for example, one or more known lubricants such as zinc stearate, lithium stearate, oleic acid, stearic amides, and ethylenebisstearamide. The amount of lubricant added is preferably 0.2% to 1% by mass based on the mass of the alloy steel powder.

Examples of other metal powders include a copper powder and a nickel powder. That is, although the present invention provides sufficient sintered compact strength without such metals, such metal powders may each be mixed in an amount of about 4% or less to achieve more advantageous properties.

It is also possible to mix, for example, alloying powders other than metal powders and powders for improving machinability. An example of an alloying powder is an iron phosphide powder, and an example of a powder for improving machinability is a manganese sulfide powder.

[0024] When lowering the oxygen content of the alloy steel powder for higher sinterability is desired, the graphite powder may be added in an amount equivalent to the sum of the amount of carbon allowed to remain in the sintered compact and the amount of carbon that reacts with oxygen in the alloy steel powder in sintering. That is, in the sintering temperature range described later, oxygen in the alloy steel powder reacts more easily with carbon than with chromium and manganese in the alloy steel powder; it binds to the carbon supplied in the form of a graphite powder and is removed as CO gas as a result of reduction. Because the amount of carbon remaining in the sintered compact is correspondingly decreased, an excess of carbon equivalent to the decrease (that is, the amount of carbon that reacts with oxygen in the alloy steel powder in sintering) may be added in advance so that the desired amount of carbon remains in the sintered compact.

[0025] It is very important to control the amount of carbon remaining in the sintered compact, that is, to determine the amount of carbon that reacts with oxygen in the alloy steel powder in advance, because the amount of carbon remaining in the sintered compact greatly affects the strength of the sintered compact. Facing this challenge, the inventors have found that the amount of graphite used for reduction of oxygen in the alloy steel powder in sintering is mainly determined by the chromium and manganese contents of the alloy steel powder and the oxidation rates of these elements, and therefore the amount of carbon allowed to remain in the sintered compact can be controlled by supplementarily adding the amount of graphite determined from the chromium and manganese contents of the alloy steel powder and the oxidation rates of these elements.

[0026] Specifically, the carbon content of the sintered compact can be controlled to the desired amount by determining the chromium content [%Cr] and the manganese content [%Mn] of the alloy steel powder and the oxidation rate α of chromium and the oxidation rate β of manganese in the alloy steel powder in advance, determining the amount of graphite powder added [%Gr] (% by mass based on the mass of the alloy steel powder for powder metallurgy) based on the amount of carbon [%C] allowed to remain in the sintered compact from equation (1) below, and adding the graphite powder to the alloy steel powder. In the present invention, therefore, the graphite powder is preferably added to the alloy steel powder for powder metallurgy in an amount [%Gr] (% by mass based on the mass of the alloy steel powder for powder metallurgy) satisfying the condition of equation (1):

[%Gr] = t × (
$$\alpha$$
 × 0.46 × [%Cr] + β × 0.29 × [%Mn]) + γ + [%C] ... (1)

wherein

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[%Cr] is the chromium content of the alloy steel powder for powder metallurgy (% by mass);

[%Mn] is the manganese content of the alloy steel powder for powder metallurgy (% by mass);

[%C] is the amount of carbon, in the graphite powder, allowed to remain in the sintered compact (% by mass based on the mass of the alloy steel powder for powder metallurgy);

 α is the oxidation rate of chromium in the alloy steel powder for powder metallurgy;

 β is the oxidation rate of manganese in the alloy steel powder for powder metallurgy;

 γ is the amount of carbon, in the graphite powder, consumed by reaction with oxygen adsorbed on the alloy steel powder for powder metallurgy and oxygen contained in the sintering atmosphere (% by mass based on the mass of the alloy steel powder for powder metallurgy), wherein $\gamma \le 0.2\%$ by mass; and t is 0.25 to 0.75.

[0027] In equation (1) above, $\alpha \times 0.46 \times [\%Cr]$ is the amount of carbon required for reduction of oxygen combined with chromium contained in the alloy steel powder, whereas $\beta \times 0.29 \times [\%Mn]$ is the amount of carbon required for reduction of oxygen combined with manganese contained in the alloy steel powder; both are intrinsic to the alloy steel powder containing chromium and manganese, which are readily oxidizable elements. Specifically, the coefficient 0.46 is calculated from the relationship 2Cr + 30 = Cr_2O_3 , where the atomic weight of chromium is 52 and the atomic weight of oxygen is 16, whereas the coefficient 0.29 is calculated from the relationship Mn + O = MnO, where the atomic weight of manganese is 55 and the atomic weight of oxygen is 16.

In equation (1) above, additionally, the upper limit of the coefficient t, namely, 0.75, is a value calculated from the mass ratio of carbon to oxygen (12:16 = 0.75:1) in the case where all oxygen combined with chromium and manganese in the

alloy steel powder is reduced under the assumption that carbon reacts with oxygen as C + O = CO. However, not all chromium oxide and manganese oxide may be reduced, depending on, for example, the sintering conditions; thus, the range of 0.25 to 0.75 is assigned to the coefficient t taking into account the amount of oxygen remaining without being reduced. The coefficient t may be limited to a narrower numerical range or a value within the range of 0.25 to 0.75 depending on the production conditions of the sintered compact (for example, t = 0.4 to 0.75, t = 0.5 to 0.75, t = 0.5, or t = 0.75).

[0028] In addition, the oxidation rate α is (the amount of chromium contained as oxide in the alloy steel powder for powder metallurgy)/(the amount of chromium in the alloy steel powder for powder metallurgy), whereas the oxidation rate β is (the amount of manganese contained as oxide in the alloy steel powder for powder metallurgy)/(the amount of manganese in the alloy steel powder for powder metallurgy). The amounts of chromium and manganese contained as oxide can be measured by dissolving the alloy steel powder for powder metallurgy into a halogen-alcohol solution prepared by dissolving, for example, bromine or iodine in an alcohol and analyzing the metal contents of extracted residue by atomic absorption spectrometry. Although no particular limitation is imposed on the oxidation rates α and P of the alloy steel powder used, oxidation rates α and β with which the value of ($\alpha \times 0.46 \times [\%Cr] + \beta \times 0.29 \times [\%Mn]$) is 0.05% by mass or more is preferred because a very clean reducing atmosphere or reduced-pressure atmosphere, which results in a cost increase, is required to lower the value of ($\alpha \times 0.46 \times [\%Cr] + \beta \times 0.29 \times [\%Mn]$) to less than 0.05% by mass.

[0029] In addition, γ is the amount of carbon, in the graphite powder added, consumed by reaction with oxygen adsorbed on the surfaces of the particles of the alloy steel powder for powder metallurgy (oxygen-containing gas and oxygen contained in the form of moisture) and oxygen contained in the sintering atmosphere (oxygen-containing gas and oxygen contained as moisture). The value of γ may be determined on the basis of an empirical rule or by examining the amount of carbon consumed when a normal pure iron powder is treated under similar compaction and sintering conditions. However, γ is 0.2% by mass or less. If γ exceeds 0.2% by mass, it is preferable to take a measure such as reducing oxygen adsorbed on the surfaces of the particles and oxygen contained in the sintering atmosphere because the sintered compact is likely to be produced under carbon-wasting conditions. On the other hand, it is generally preferable that γ be 0.01% by mass or more because lowering γ to less than 0.01% by mass requires strict oxidation control throughout the entire process to sintering, which affects the production costs.

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The tolerance of the amount of graphite powder actually added with respect to the target [%Gr] may be set depending on the purpose. For example, the tolerance may be [%Gr] \pm 0.05% by mass. Alternatively, [%Gr] may be rounded in predetermined steps, for example, in steps of 0.05% by mass or 0.1% by mass. On the other hand, it is also possible to set [%Gr] in steps of 0.01% by mass or less.

With the amount of graphite powder determined by equation (1) above, the amount of graphite-derived carbon remaining in the sintered compact can at least be controlled approximately to the range of $[\%C] \pm 0.05\%$ by mass.

[0030] The "alloy steel powder or mixed powder containing the alloy steel powder" to be compacted under pressure and sintered is hereinafter referred to as "raw material powder" for convenience of description.

Although no particular limitation is imposed on, for example, the configuration of the equipment used for compacting the raw material powder under pressure, it must be compacted at a pressure of 700 MPa or more. If the compaction pressure falls below 700 MPa, a sintered compact having sufficient strength cannot be achieved. In addition, because the resulting compact has insufficient density if the compaction pressure falls below 700 MPa, carburizing and quenching treatment after sintering (generally, treatment for heating a sintered compact in a carburizing gas atmosphere to dissolve (carburize) carbon into the sintered compact and then quenching it in oil) involves intergranular oxidation due to oxygen contained in the carburizing gas atmosphere combines with chromium and manganese, which are readily oxidizable elements, in the sintered compact), thus decreasing the strength of the sintered compact (heat-treated compact). In contrast, if the compaction is performed at a compaction pressure of 700 MPa or more, a compact having sufficient density can be achieved, and accordingly a heat-treated compact having sufficient strength can be achieved because intergranular oxidation is inhibited during carburizing and quenching treatment after sintering.

In addition, the compaction is preferably performed at room temperature (about 20°C) to 160°C. For example, good compressibility is achieved by charging a raw material powder at room temperature and compacting it under pressure while maintaining the die temperature to 50°C to 70°C. It is also possible to apply a compaction technique in which the die and the raw material powder are heated to 120°C to 130°C (so-called warm compaction).

[0031] The sintering after the compaction must be performed at a temperature of 1,150°C to 1,300°C. If the sintering temperature falls below 1,150°C, a sintered compact having sufficient strength cannot be achieved because the powder particles are insufficiently bonded by insufficient above-mentioned reduction reaction of oxygen. On the other hand, if the sintering temperature exceeds 1,300°C, the crystal grains become coarse, thus decreasing the strength. In particular, the sintering temperature is preferably 1,150°C to 1,200°C in view of production costs.

In addition, the sintering time for the above sintering temperature is preferably about 20 to 120 minutes in view of sinterability and production costs.

[0032] In addition, the sintering atmosphere used is, for example, a reducing gas, an inert gas, or a hydrocarbon-converted gas (i.e., RX gas). Alternatively, the atmosphere may be in a vacuum. The alloy steel powder used in the present invention contains reduced amounts of chromium and molybdenum, which are readily oxidizable elements, so that intergranular oxidation is inhibited during sintering even in an RX atmosphere, thus providing sufficient properties to serve as, for example, an automobile high-strength sintered part.

Although no particular limitation is imposed on, for example, the sintering equipment used, a mesh belt furnace or a pusher furnace is preferably used in view of reducing the sintering costs because they offer mass production.

Although no particular limitation is imposed on the oxygen content of the resulting sintered compact, it is generally preferable that the oxygen content be lowered to 0.1% by mass or less, more preferably 0.05% by mass or less. In addition, although no particular limitation is imposed on the carbon content of the sintered compact, the carbon content is generally about 0.1% to 0.9% by mass.

[0033] The sintered compact thus obtained can be used as-sintered as an automobile high-strength sintered part. Optionally, however, heat treatment such as carburizing and quenching (i.e., CQT), bright quenching (i.e., BQT), high-frequency quenching, or carbonitriding heat treatment may be performed. If carburizing and quenching, bright quenching, or high-frequency quenching is performed, that treatment is preferably followed by tempering. Such heat treatment further improves the properties to serve as an automobile high-strength sintered part. In the heat treatment, no particular limitation is imposed on the operating conditions and the configuration of the equipment used, and a known technique may be used.

20 EXAMPLES

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EXAMPLE 1

[0034] The raw material powders used were mixtures of alloy steel powders having the compositions shown in Table 1 and a graphite powder added in such amounts that the carbon content after sintering (the carbon content of a sintered compact) was 0.3% by mass. These raw material powders were compacted into rectangular-column compacts having a bottom surface measuring 10 mm \times 60 mm at a compaction pressure of 700 MPa and were sintered at 1,200°C in a nitrogen gas atmosphere. After the rectangular compacts were subjected to carburizing, quenching, and tempering, the tensile strengths and impact values thereof were measured, and the metallographic structures thereof were observed. In addition, the sintered compacts were analyzed for carbon content before the carburizing, quenching, and tempering. The results are shown in Table 1.

[0035] In Table 1, the sintered compact of Sample No. 1 (comparative example), in which the chromium content of the alloy steel powder was low, had low tensile strength because the amount of martensite formed was small. On the other hand, the sintered compact of Sample No. 4 (comparative example), in which the chromium content was excessive, had low tensile strength because intergranular oxidation occurred. In contrast, the sintered compacts of Sample Nos. 2 and 3, which are invention examples, had high tensile strengths, namely, not less than 1,000 MPa, because sufficient quench hardenability was ensured.

In addition, the sintered compact of Sample No. 5 (comparative example), in which the manganese content of the alloy steel powder was low, had low tensile strength because the amount of martensite formed was small. On the other hand, the sintered compact of Sample No. 8 (comparative example), in which the manganese content was excessive, had low tensile strength because intergranular oxidation occurred. In contrast, the sintered compacts of Sample Nos. 6 and 7, which are invention examples, had high tensile strengths, namely, not less than 1,000 MPa, because sufficient quench hardenability was ensured.

[0036] In addition, the sintered compact of Sample No. 9 (comparative example), in which the molybdenum content of the alloy steel powder was low, had low tensile strength because the amount of martensite formed was small. On the other hand, the sintered compact of Sample No. 12 (comparative example), in which the molybdenum content was excessive, had low tensile strength because the raw material compact had decreased compressibility. In contrast, the sintered compacts of Sample Nos. 10 and 11, which are invention examples, had high tensile strengths, namely, not less than 1,000 MPa, because sufficient quench hardenability was ensured.

In addition, the sintered compact of Sample No. 16 (comparative example), in which the oxygen content of the alloy steel powder was excessive, had low tensile strength because the raw material compact had decreased compressibility. In contrast, the sintered compacts of Sample Nos. 13 to 15, which are invention examples, had high tensile strengths, namely, not less than 1,000 MPa.

[0037] [Table 1]

Table 1

	Sample	Com	position		y steel ass)	powder	(% by	Amount of graphite	Carbon content of sintered	Tensile	Impact	
5	No.	Fe	С	Cr	Mn	Мо	0	added (% by mass)*1	compact (% by mass)	strength (MPa)	value (J/cm ²)	Category
10	1	Bal.	0.004	0.2	0.3	0.2	0.3	0.60	0.30	700	11	Comparative example
	2	Bal.	0.004	0.3	0.3	0.2	0.3	0.60	0.30	1060	9	Invention example
15	3	Bal.	0.004	0.7	0.3	0.2	0.3	0.60	0.30	1050	9	Invention example
	4	Bal.	0.004	8.0	0.3	0.2	0.3	0.60	0.30	750	7	Comparative example
20	5	Bal.	0.004	0.5	0.05	0.2	0.3	0.60	0.30	710	10	Comparative example
	6	Bal.	0.004	0.5	0.1	0.2	0.3	0.60	0.30	1040	9	Invention example
25	7	Bal.	0.004	0.5	0.5	0.2	0.3	0.60	0.30	1000	9	Invention example
	8	Bal.	0.004	0.5	0.55	0.2	0.3	0.60	0.30	680	6	Comparative example
30	9	Bal.	0.004	0.5	0.3	0.05	0.3	0.60	0.30	630	12	Comparative example
	10	Bal.	0.004	0.5	0.3	0.1	0.3	0.60	0.30	1070	9	Invention example
35	11	Bal.	0.004	0.5	0.3	0.5	0.3	0.60	0.30	1080	9	Invention example
	12	Bal.	0.004	0.5	0.3	0.55	0.3	0.60	0.30	850	7	Comparative example
40	13	Bal.	0.004	0.5	0.3	0.2	0.25	0.55	0.30	1060	10	Invention example
40	14	Bal.	0.004	0.5	0.3	0.2	0.35	0.65	0.30	1050	9	Invention example
45	15	Bal.	0.004	0.5	0.3	0.2	0.5	0.80	0.30	1000	9	Invention example
45	16	Bal.	0.004	0.5	0.3	0.2	0.6	0.90	0.30	860	7	Comparative example
	*1 % by m	ass ba	sed on r	mass o	of alloy	steel po	wder					

EXAMPLE 2

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[0038] The raw material powder used was a mixture of the alloy steel powder used for Sample No. 2 in Example 1 (Table 1) (containing 0.3% by mass of chromium, 0.3% by mass of manganese, 0.2% by mass of molybdenum, 0.004% by mass of carbon, and 0.3% by mass of oxygen, the balance being iron and incidental impurities), a graphite powder added in such an amount that the carbon content after sintering (the carbon content of a sintered compact) was 0.3% by mass, and a compaction lubricant (zinc stearate). This raw material powder was compacted into rectangular-column compacts having a bottom surface measuring 10 mm \times 60 mm and was sintered in a nitrogen gas atmosphere. After

the rectangular compacts were subjected to carburizing, quenching, and tempering, the tensile strengths and densities thereof were measured. The results are shown in Table 2 together with the compaction pressure and the sintering temperature. In addition, Fig. 1 contrasts the relationships between compaction pressure and tensile strength of Sample Nos. 2 and 5 to 7 with those of Sample Nos. 8 to 11, which are existing high-strength materials.

[0039] In Table 2, the sintered compact of Sample No. 1 (comparative example), in which the sintering temperature was insufficient, had low tensile strength because the particles of the alloy steel powder were insufficiently bonded and intergranular oxidation occurred during carburizing and quenching. On the other hand, the sintered compact of Sample No. 4 (comparative example), in which the sintering temperature was excessive, had low tensile strength because the crystal grains became coarse. Additionally, as shown in Fig. 1, the sintered compacts of Sample Nos. 5 and 6 (comparative examples), in which the pressure for compaction was insufficient, had significantly lower tensile strengths than the existing high-strength materials (Sample Nos. 8 to 11) because the low densities thereof resulted in intergranular oxidation occurred during carburizing and quenching. In contrast, the sintered compacts of Sample Nos. 2, 3, and 7, which are invention examples, had high tensile strengths, namely, not less than 1,000 MPa.

[0040] [Table 2]

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Table 2

Sample No.	Compaction pressure (MPa)	Sintering temperature (°C)	Density (g/cc)	Tensile strength (MPa)	Category
1	700	1130	7.16	850	Comparative example
2	700	1150	7.18	1050	Invention example
3	700	1300	7.20	1070	Invention example
4	700	1330	7.22	960	Comparative example
5	500	1150	6.91	630	Comparative example
6	600	1150	7.09	760	Comparative example
7	800	1150	7.26	1140	Invention example
8	500	1130	6.93	850	Conventional example *1
9	600	1150	7.12	935	Conventional example *1
10	700	1300	7.20	1005	Conventional example *1
11	800	1350	7.26	1100	Conventional example *1

EXAMPLE 3

[0041] The raw material powders used were mixtures of alloy steel powders having the compositions shown in Table 3 and a graphite powder. These raw material powders were compacted into rectangular-column compacts having a bottom surface measuring 10 mm \times 60 mm at a compaction pressure of 700 MPa and were sintered at 1,200°C in a nitrogen gas atmosphere. The tensile strengths of the rectangular compacts were measured, and they were analyzed for carbon content. The results are shown in Table 3.

In Table 3, the sintered compacts of Sample Nos. 1 to 4, in which the amount of graphite powder added was fixed at 0.60% by mass, had variations in carbon content and therefore had large variations in tensile strength because the amount of carbon removed increased with increasing oxygen content of the alloy steel powders. On the other hand, the sintered compacts of Sample Nos. 5 to 7, in which the graphite powder was added taking into account the amount of carbon that reacts with oxygen in sintering, had no substantial variations in carbon content and tensile strength.

[0042] [Table 3]

Table 3

	Comp	osition of a	alloy ste	el powd	er (% by	mass)		Carbon	Tensile strength (MPa)	
Sample No.	Fe	С	Cr	Mn	Мо	0	Amount of graphite added (% by mass)*1	content of sintered compact (% by mass)		
1	Bal.	0.005	0.5	0.3	0.2	0.25	0.60	0.34	348	
2	Bal.	0.005	0.5	0.3	0.2	0.35	0.60	0.24	305	
3	Bal.	0.005	0.5	0.3	0.2	0.45	0.60	0.16	260	
4	Bal.	0.005	0.5	0.3	0.2	0.5	0.60	0.11	220	
5	Bal.	0.005	0.5	0.3	0.2	0.35	0.70	0.34	347	
6	Bal.	0.005	0.5	0.3	0.2	0.45	0.80	0.34	345	
7	Bal.	0.005	0.5	0.3	0.2	0.5	0.85	0.34	342	
*1 % by mass	based	on mass	of alloy	steel po	wder		·			

EXAMPLE 4

[0043] The raw material powders used were mixtures of alloy steel powders having the compositions shown in Table 4 and a graphite powder added in amounts determined by equation (1) above (t = 0.75, γ = 0.1% by mass) so that the carbon content after sintering (the carbon content of a sintered compact) was 0.4% by mass. These raw material powders were compacted into rectangular-column compacts having a bottom surface measuring 10 mm \times 60 mm at a compaction pressure of 700 MPa and were sintered at 1,200°C in a nitrogen gas atmosphere. After the rectangular compacts were subjected to carburizing, quenching, and tempering, the tensile strengths and impact values thereof were measured, and the metallographic structures thereof were observed. In addition, the sintered compacts were analyzed for carbon content before the carburizing, quenching, and tempering. The results are shown in Table 4.

According to Table 4, the carbon contents of all sintered compacts (excluding the sintered compact of No. 16) measured by carbon content analysis were nearly 0.40% by mass (±0.03% by mass), demonstrating that the amount of graphite powder added can be accurately determined depending on the target carbon content of a sintered compact by equation (1) above. The carbon content of the sintered compact of No. 16, in which the oxygen content of the alloy steel powder was high, deviated largely from the target $\alpha\alpha\alpha$ value (0.40% by mass) based on equation (1), probably because γ deviated considerably from the value assigned to equation (1), namely, 0.1% by mass.

[0044] [Table 4]

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Table 4

		Comp	osition	of iron-based al	loy steel	powder (% by	mass)			Carbon			
Sample No.	Fe	С	Cr	Oxidation rate α	Mn	Oxidation rate β	Мо	0	Amount of graphite added (% by mass)*1	content of sintered compact (% by mass)	Tensile strength (MPa)	Impact value (J/cm ²)	Category
1	Bal.	0.004	0.2	0.85	0.2	0.95	0.2	0.20	0.60	0.41	750	12	Comparative example
2	Bal.	0.004	0.3	0.85	0.2	0.95	0.2	0.26	0.63	0.40	1100	10	Invention example
3	Bal.	0.004	0.7	0.85	0.2	0.95	0.2	0.35	0.75	0.40	1090	10	Invention example
4	Bal.	0.004	0.8	0.85	0.2	0.95	0.2	0.51	0.78	0.39	790	8	Comparative example
5	Bal.	0.004	0.5	0.85	0.05	0.95	0.2	0.28	0.66	0.41	720	10	Comparative example
6	Bal.	0.004	0.5	0.85	0.1	0.95	0.2	0.29	0.67	0.41	1050	9	Invention example
7	Bal.	0.004	0.5	0.85	0.5	0.95	0.2	0.41	0.75	0.39	1010	9	Invention example
8	Bal.	0.004	0.5	0.85	0.55	0.95	0.2	0.51	0.76	0.38	690	6	Comparative example
9	Bal.	0.004	0.5	0.85	0.2	0.95	0.05	0.32	0.69	0.40	670	13	Comparative example
10	Bal.	0.004	0.5	0.85	0.2	0.95	0.1	0.32	0.69	0.40	1110	10	Invention example
11	Bal.	0.004	0.5	0.85	0.2	0.95	0.5	0.32	0.69	0.40	1020	10	Invention example
12	Bal.	0.004	0.5	0.85	0.2	0.95	0.55	0.32	0.69	0.40	890	8	Comparative example

(continued)

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		Comp	osition	of iron-based al	loy steel	powder (% by	mass)			Carbon			
Sample No.	Fe	С	Cr	Oxidation rate α	Mn	Oxidation rate β	Мо	0	Amount of graphite added (% by mass)*1	content of sintered compact (% by mass)	Tensile strength (MPa)	Impact value (J/cm ²)	Category
13	Bal.	0.004	0.5	0.70	0.2	0.90	0.2	0.28	0.66	0.41	1100	11	Invention example
14	Bal.	0.004	0.5	0.90	0.2	0.97	0.2	0.33	0.70	0.40	1090	10	Invention example
15	Bal.	0.004	0.5	0.95	0.2	0.99	0.2	0.40	0.71	0.37	1040	10	Invention example
16	Bal.	0.004	0.5	1.00	0.2	1.00	0.2	0.60	0.72	0.19	890	8	Comparative example
<u> </u>											_		

^{*1 %} by mass based on mass of alloy steel powder

EXAMPLE 5

[0045] The raw material powders used were mixtures of alloy steel powders having the compositions shown in Table 5 and a graphite powder. These raw material powders were compacted into rectangular-column compacts having a bottom surface measuring 10 mm x 60 mm at a compaction pressure of 700 MPa and were sintered at 1,200°C in a nitrogen gas atmosphere. The tensile strengths of the rectangular compacts were measured, and they were analyzed for carbon content. The results are shown in Table 5.

In Table 5, the sintered compacts of Sample Nos. 1 to 4, in which the amount of graphite powder added was fixed at 0.60% by mass, had variations in carbon content and therefore had large variations in tensile strength because the amount of carbon removed increased with increasing oxygen content of the alloy steel powders. On the other hand, the sintered compacts of Sample Nos. 5 to 7, in which the graphite powder was added in amounts determined taking into account the amount of carbon that reacts with oxygen in sintering by equation (1) above (t = 0.75, γ = 0.1% by mass), had no substantial variations in carbon content and tensile strength.

[0046] Table 5

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Table 5

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	(Compositi	ion of i	ron-based all	oy stee	el powder (%	by mas	s)	Amount of	Carbon	
Sample No.	Fe	С	Cr	Oxidation rate α	Mn	Mn Oxidation rate β	Мо	0	graphite added (% by mass)*1	content of sintered compact (% by mass)	Tensile strength (MPa)
1	Bal.	0.005	0.5	0.40	0.2	0.55	0.2	0.25	0.60	0.41	360
2	Bal.	0.005	0.5	0.60	0.2	0.70	0.2	0.28	0.60	0.37	320
3	Bal.	0.005	0.5	0.80	0.2	0.85	0.2	0.33	0.60	0.32	275
4	Bal.	0.005	0.5	1.00	0.2	1.00	0.2	0.39	0.60	0.25	235
5	Bal.	0.004	0.5	0.60	0.2	0.70	0.2	0.28	0.63	0.41	360
6	Bal.	0.004	0.5	0.80	0.2	0.85	0.2	0.33	0.67	0.40	355
7	Bal.	0.004	0.5	1.00	0.2	1.00	0.2	0.39	0.72	0.39	345
*1 % by m	ass bas	sed on ma	ass of	alloy steel po	wder	•	•	•	•	•	

EXAMPLE 6

[0047] The raw material powders used were mixtures of alloy steel powders having the compositions shown in Table 6 and a graphite powder. These raw material powders were compacted into rectangular-column compacts having a bottom surface measuring 10 mm \times 60 mm at a compaction pressure of 700 MPa and were sintered at 1,150°C in a nitrogen gas atmosphere. The tensile strengths of the rectangular compacts were measured, and they were analyzed for carbon content. The results are shown in Table 6.

In Table 6, the sintered compacts of Sample Nos. 1 to 3, in which the graphite powder was added in amounts determined taking into account the amount of carbon that reacts with oxygen in sintering by equation (1) above (t = 0.5, $\gamma = 0.1\%$ by mass) so that the carbon contents of the sintered compacts were 0.5% by mass, had no substantial variations in carbon content and tensile strength.

[0048] Table 6

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Table 6

	(Compositi	on of i	ron-based all	oy stee	l powder (%	by mas	s)	Amount of	Carbon	
Sample No.	Fe	Fe C Cr		Oxidation rate α	Mn	Oxidation rate β	Мо	0	graphite added (% by mass)*1	content of sintered compact (% by mass)	Tensile strength (MPa)
1	Bal.	0.004	0.5	0.60	0.2	0.70	0.2	0.28	0.70	0.51	350
2	Bal.	0.004	0.5	0.80	0.2	0.85	0.2	0.33	0.72	0.50	345
3	Bal.	0.004	0.5	1.00	0.2	1.00	0.2	0.39	0.73	0.49	345
*1 % by m	nass ba	sed on n	nass (of alloy steel	powde	er					

Industrial Applicability

[0049] According to the present invention, a high-strength sintered compact that can be used as an automobile high-strength sintered part can be produced at low cost by a method for producing a sintered compact by powder metallurgy using an alloy steel powder containing no nickel or copper and containing a relatively small amount of molybdenum.

Claims

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- 1. A method for producing a sintered compact by powder metallurgy, comprising compacting an alloy steel powder for powder metallurgy or a mixed powder containing the alloy steel powder at a pressure of 700 MPa or more and sintering the compact at a temperature of 1,150°C to 1,300°C, wherein the alloy steel powder used contains 0.3% to 0.7% by mass of chromium, 0.1% to 0.5% by mass of manganese, 0.1% to 0.5% by mass of molybdenum, and 0.25% to 0.5% by mass of oxygen, the balance being iron and incidental impurities.
 - 2. The method for producing a sintered compact according to Claim 1, wherein the mixed powder contains a graphite powder.
 - 3. The method for producing a sintered compact according to Claim 1 or 2, wherein the mixed powder contains a graphite powder in an amount equivalent to the sum of the amount of carbon allowed to remain in the sintered compact and the amount of carbon that reacts with oxygen in the alloy steel powder for powder metallurgy in the sintering.
- 4. The method for producing a sintered compact according to Claim 2, wherein the graphite powder is added to the alloy steel powder for powder metallurgy in an amount [%Gr] (% by mass based on the mass of the alloy steel powder for powder metallurgy) satisfying the condition of equation (1):

[%Gr] = t × (
$$\alpha$$
 × 0.46 × [%Cr] + β × 0.29 × [%Mn]) + γ + [%C] ... (1)

wherein

[%Cr] is the chromium content of the alloy steel powder for powder metallurgy (% by mass);

[%Mn] is the manganese content of the alloy steel powder for powder metallurgy (% by mass);

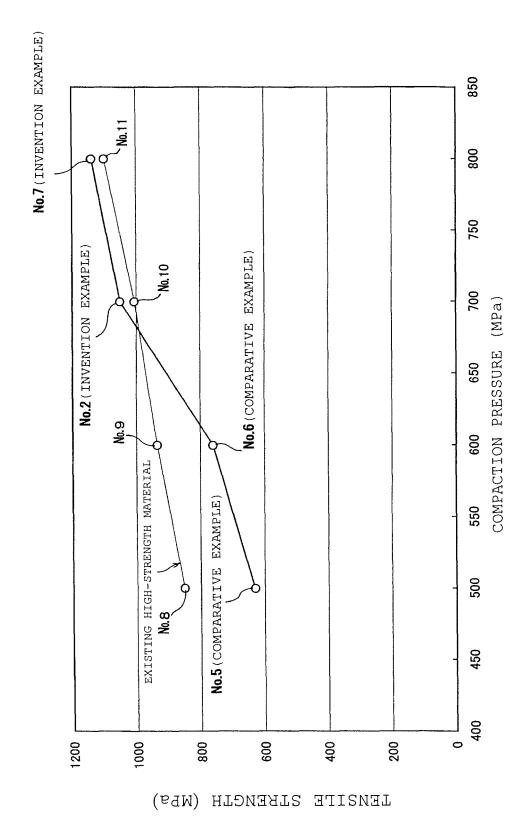
[%C] is the amount of carbon, in the graphite powder, allowed to remain in the sintered compact (% by mass based on the mass of the alloy steel powder for powder metallurgy);

 α is the oxidation rate of chromium in the alloy steel powder for powder metallurgy;

 β is the oxidation rate of manganese in the alloy steel powder for powder metallurgy;

 γ is the amount of carbon, in the graphite powder, consumed by reaction with oxygen adsorbed on the alloy steel powder for powder metallurgy and oxygen contained in a sintering atmosphere (% by mass based on the mass of the alloy steel powder for powder metallurgy), wherein $\gamma \le 0.2\%$ by mass; and t is 0.25 to 0.75.

FIG.1



INTERNATIONAL SEARCH REPORT

International application No.

	PCT/JP2009/06	6865						
A. CLASSIFICATION OF SUBJECT MATTER <i>C22C33/02</i> (2006.01) i, <i>C22C38/00</i> (2006.01) :	i							
According to International Patent Classification (IPC) or to both national	al classification and IPC							
B. FIELDS SEARCHED								
Minimum documentation searched (classification system followed by cl B22F1/00-B22F8/00, C22C1/04, C22C1/05, C22C1/05, C22C1/04, C22C1/05, C22C1/05								
Kokai Jitsuyo Shinan Koho 1971-2009 To	tsuyo Shinan Toroku Koho 1996-200 proku Jitsuyo Shinan Koho 1994-200	09						
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Further documents are listed in the continuation of Box C.	See patent family annex.							
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Date of the actual completion of the international search 19 November, 2009 (19.11.09)	Date of mailing of the international search report 01 December, 2009 (01.12.0)9)						
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INTERNATIONAL SEARCH REPORT

International application No.
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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
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