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(54) Title: LOW ALLOYED STEEL POWDER

(57) Abstract: A water atomised prealloyed iron-based steel powder which comprises by weight-%: 0.4-2.0 Cr, 0.1-0.8 Mn, less than 0.1 V, less than 0.1 Mo, less than 0.1 Ni, less than 0.2 Cu, less than 0.1 C, less than 0.25 O, less than 0.5 of unavoidable impurities, and the balance being iron.

LOW ALLOYED STEEL POWDER

FIELD OF THE INVENTION

The present invention concerns a low alloyed iron-based powder, a powder composition containing the powder and other additives, and a component made by compaction and sintering of the iron-base powder composition containing the new low alloyed steel powder. The mechanical properties of the component made from the invented powder are comparable with the mechanical properties of a component made from a more highly alloyed, and more expensive diffusion bonded powder.

BACKGROUND OF THE INVENTION

In industries the use of metal products manufacturing by compaction and sintering metal powder compositions is becoming increasingly widespread. A number of different products of varying shape and thickness are being produced and the quality requirements are continuously raised at the same time as it is desired to reduce the cost. As net shape components, or near net shape components requiring a minimum of machining in order to reach finished shape, are obtained by pressing and sintering of iron powder compositions in combination with a high degree of material utilisation, this technique has a great advantage over conventional techniques for forming metal parts such as moulding or machining from bar stock or forgings.

One problem connected to the press and sintering method is however that the sintered component contains a certain amount of pores decreasing the strength of the component. Basically there are two ways to overcome the negative effect on mechanical properties caused by the component porosity. 1) The strength of the sintered component may by increased by introducing alloying elements such as carbon, copper, nickel molybdenum etc. 2) The porosity of the sintered component may be reduced by increasing the compressibility of the powder composition, and/or increasing the compaction pressure for a higher green density, or increasing the shrinkage of the component during sintering. In practise a combination of strengthening the component by addition of alloying elements and minimising the porosity are applied. Thus, various compositions of low-alloyed steel powders, and methods for compaction of these powders are known for production of PM component showing high strength and hardness. However, a characteristic property of PM components is a relative low toughness compared to wrought steel materials. The so called diffusion alloyed iron based powders, having a relatively high compressibility despite being “highly” alloyed, provides possibilities for producing compacted and sintered bodies having a high toughness and high elongation in combination with a high strength compared to prealloyed powders.

However, a drawback related to presently used diffusion alloyed powders is their relatively high content of costly alloying elements such as molybdenum and nickel. It has now unexpectedly been found that by a careful selection of a combination of the alloying elements chromium and manganese, at relatively low content, a prealloyed powder is obtained giving a pressed and sintered body mechanical properties with respect to elongation and strength at the same level or close to the values which can be obtained by using a more alloyed diffusion bonded powder.

US 4 266 974 discloses examples of alloyed powders outside the claimed scope containing only manganese and chromium as intentionally added alloying elements. The examples contains 2.92 % of chromium in combination with 0.24 % of manganese, 4.79 % of chromium in combination with 0.21 % by weight of manganese or 0.55 % of chromium in combination with 0.89 % by weight of manganese.

In Japanese patent publication number JP59173201 a method of reduction annealing of a low alloyed steel powder containing chromium, manganese and molybdenum, one example shows a powder having a chromium content of 1.14 % by weight and a manganese content of 1.44 % by weight as the only intentionally added alloying elements.

A chromium, manganese and molybdenum based pre-alloyed steel powder is described in US 6 348 080. WO03/106079 teaches a chromium, manganese and molybdenum alloyed steel powder having lower content of alloying elements compared the steel powder described in US 6 348 080. The powder is suitable to form bainitic structures at a carbon content above about 0.4 % by weight.

OBJECTS OF THE INVENTION

An object of the invention is to provide an alloyed iron-based powder suitable for producing compacted and sintered components, the powder being essentially free from costly alloying elements such as molybdenum and nickel.

Another object of the invention is to provide a powder capable of forming compacted and sintered components having good elongation, tensile strength and yield strength.

Another object of the invention is to provide a sintered part having the above mentioned properties.

SUMMARY OF THE INVENTION

At least one of these objects is accomplished by:

- A water atomised prealloyed iron-based steel powder which comprises by weight-%: 0.4-2.0 Cr, 0.1-0.8 Mn, less than 0.1 V, less than 0.1 Mo, less than 0.1 Ni, less than 0.2 Cu, less than 0.1 C, less than 0.25 O, less than 0.5 of unavoidable impurities, and the balance being iron.

- An iron-based powder composition based on the steel powder and mixed with 0.35-1 % by weight of the composition of graphite, 0.05-2 % by weight of the composition of lubricants and optionally copper in an amount up to 3 %, hard phase materials and machinability enhancing agents.

- A method of producing a sintered component comprising the steps of;
  a) preparing the iron-based steel powder composition based on the steel powder,
  b) subjecting the composition to compaction between 400 and 2000 MPa,
  c) sintering the obtained green component in a reducing atmosphere at temperature between 1000-1400 °C,
  d) optionally forging the heated component at a temperature above 500 °C or subjecting the obtained sintered component to a heat treatment or hardening step.

- A sintered component produced by the method having a pearlitic/ferritic microstructure

The steel powder has low and defined contents of chromium and manganese and is essentially free from molybdenum, nickel and vanadium.

DETAILED DESCRIPTION OF THE INVENTION

Preparation of the iron-based alloyed steel powder.

The steel powder is produced by water atomization of a steel melt containing defined amounts of alloying elements. The atomized powder is further subjected to a reduction annealing process such as described in the US patent 6,027,544, herewith incorporated by reference. The particle size of the steel powder could be any size as long as it is compatible with the press and sintering or powder forging processes. Examples of suitable particle size is the particle size of the known powder ABC100.30 available from Höganäs AB, Sweden, having about 10 % by weight above 150 μm and about 20 % by weight below 45 μm .

<u>Contents of the steel powder</u>

Chromium serves to strengthen the matrix by solid solution hardening. Furthermore, chromium will increase the hardenability, oxidation resistance and abrasion resistance of the sintered body. A content of chromium above 2.0 wt% will however reduce the compressibility of the steel powder and render the formation of a ferritic/pearlitic microstructure more difficult. Preferably from the viewpoint of compressibility the maximum content is about 1.8 wt%, even more preferred 1.5 wt%. A Cr content below 0.4 % by weight will have insignificant effect on desired properties. Preferably, the chromium content is at least 0.5 wt%.

Manganese will, as for chromium, increase the strength, hardness and hardenability of the steel powder. A content above 0.8 wt% will increase the formation of manganese containing inclusion in the steel powder and will also have a negative effect on the compressibility due to solid solution hardening and increased ferrite hardness. Preferably, the manganese content is below 0.7 wt%, even more preferably the manganese content is below 0.6 wt%. If the manganese content is below 0.1 % desired properties cannot be obtained, furthermore, it will not possible to use recycled scrap unless a specific treatment for the reduction during the course of the steel manufacturing is carried out, For these reasons the manganese content is preferably at least 0.2 wt%, even more preferred 0.3 wt%. Thus the manganese content should be between 0.1-0.8 wt%, preferably 0.2-0.7 wt%, even more preferred 0.3-0.6 wt%.

It has also been found that in order to obtain a sufficiently high compressibility the total amount of chromium and manganese, which to some extent are exchangeable with each other, should not be more than 2.5 % by weight, preferably not more than 2.3 % by weight, most preferably not more than 2.0 % by weight.

In one embodiment with low chromium contents in the range of 0.4-0.6 wt% Cr, the low chromium content is compensated by a comparably high manganese content in the range of 0.6-0.8 wt%, preferably 0.7-0.8 wt%. This is embodiment is advantageous since manganese is less expensive than chromium.

In another embodiment, when the chromium content is at least 0.7 wt% the manganese content is at most 0.5 wt%, and when the chromium content is at least 1.0 wt% the manganese content is at most 0.4 wt%, preferably at most 0.3 wt%. By having high chromium content, the manganese content may be kept lower, thereby minimising any formation of manganese containing inclusion in the steel powder.

Oxygen suitably is at most 0.25 wt%, to prevent formation of oxides with chromium and manganese that impairs strength and compressibility of the powder. For these reasons oxygen preferably is at most 0.18 wt%.

Vanadium and nickel should be less than 0.1 wt% and copper less than 0.2 wt%. A too high content of these elements will have a negative effect on compressibility and may increase costs. Also, the presence of nickel will suppress ferrite formation, thus promoting a brittle pearlitic/bainitic structure. Molybdenum should be less than 0.1 wt% to prevent bainite to be formed as well as to keep costs low since molybdenum is a very expensive alloying element.

Carbon in the steel powder shall be at most 0.1 % by weight and oxygen at most 0.25 % by weight. Higher contents will unacceptably reduce the compressibility of the powder. For the same reason nitrogen shall be kept less than 0.1 wt%.

The total amount of inevitable impurities should be less than 0.5 % by weight in order not to impair the compressibility of the steel powder or act as formers of detrimental inclusions.

Iron-based powder composition

Before compaction the iron-based steel powder is mixed with graphite and lubricants. Graphite is added in an amount between 0.35-1.0 % by weight of the composition and lubricants are added in an amount between 0.05-2.0 % by weight of the composition. In a certain embodiment copper in the form of copper powder may be added in an amount up to 3 % by weight. In another embodiment nickel powder up to 5% by weight with or without additional copper powder may be added to the composition by admixing.

Amount of graphite

In order to enhance strength and hardness of the sintered component carbon is introduced in the matrix. Carbon is added as graphite in amount between 0.35-1.0 % by weight of the composition. An amount less than 0.35 % by weight will result in a too low strength and an amount above 1.0 % will result in an excessive formation of carbides yielding a too high hardness, insufficient elongation and impair the machinability properties. In case that after sintering or forging, the component is heat treated with a heat treatment process including carburising; the amount of added graphite may be less than 0.35 %.

Amount of copper

Copper is a commonly used alloying element in the powder metallurgical technique. Copper will enhance the strength and hardness through solid solution hardening. Copper will also facilitate the formation of sintering necks during sintering as copper melts before the sintering temperature is reached providing so called liquid phase sintering which is much faster than sintering in solid state. In a certain embodiment copper may be added in an amount up to 3% by weight.

Amount o nickel

Nickel is a commonly used alloying element in the powder metallurgical technique. Nickel will enhance the strength and hardness through solid solution hardening. Nickel will also strengthen the sintering necks during sintering. In a certain embodiment nickel may be added in an amount up to 5% by weight.

Amount of lubricants

Lubricants are added to the composition in order to facilitate the compaction and ejection of the compacted component. The addition of less than 0.05 % by weight of the composition of lubricants will have insignificant effect and the addition of above 2 % by weight of the composition will result in a too low density of the compacted body. Lubricants may be chosen from the group of metal stearates, waxes, fatty acids and derivates thereof, oligomers, polymers and other organic substances having lubricating effect.

Other substances

Other substances such as hard phase materials and machinability enhancing agents, such as MnS, MoS_2, CaF_2, and different kinds of minerals etc. may be added.

<u>Sintering</u>

The iron-based powder composition is transferred into a mould and subjected to a compaction pressure of about 400-2000 MPa to a green density of above about 6.75

g/cm^3. The obtained green component is further subjected to sintering in a reducing atmosphere at a temperature of about 1 000-1 400° C, preferably between about 1100-1300° C.

Post sintering treatments

The sintered component may be subjected to a hardening process for obtaining desired microstructure through heat treatment including cooling at a controlled cooling rate. The hardening process may include known processes such as case hardening, nitriding, induction hardening and the like. In case that heat treatment includes carburising the amount of added graphite may be less than 0.35 %.

Alternatively, the sintered component may be subjected to a forging operation in order to reach full density. The forging operation may be performed either directly after the sintering operation when the temperature of the component is about 500-1 400° C, or after cooling of the sintered component, the cooled component is then reheated to a temperature of about 500-1 400° C prior to the forging operation.

Other types of post sintering treatments may utilized such as surface rolling or shot peening which introduces compressive residual stresses enhancing the fatigue life.

Properties of the finished component

The present invention provides a new iron-based prealloyed powder for the manufacture of sintered components having tensile strength and elongation comparable with the corresponding values obtained from a diffusion bonded powder containing higher total amount of alloying elements, and more expensive alloying elements such as nickel and molybdenum. Especially, the present invention provides a chromium and manganese pre-alloyed iron-based powder, a composition containing the powder, as well as a compacted and sintered component made from the powder composition. The compacted and sintered component exhibits a value for elongation above 2 % in combination with a yield strength of about 500 MPa. The microstructure is pearlitic or pearlitic/ferritic.

EXAMPLES

Various prealloyed iron-based steel powders 1-5 were produced by water atomizing of steel melts. The obtained raw powders were further annealed in a hydrogen atmosphere at 1160°C followed by gently grinding to disintegrate the sintered powder cake. The particle size of the powders was below 150 μm. Table 1 shows the chemical compositions of the different powders. Powder 6 was DISTALOY AB, a commercial diffusion-alloyed powder available from Höganäs, Sweden, and based on the high-purity atomised powder ASC100.29 (plain iron).

Table 1

Powder	Cr [%]	Mn [%]	V [%]	Cu [%]	Mo [%]	Ni [%]	C [%]	O [%]
1	0.80	0.35	-	-	-	-	0.003	0.090
2	0.68	0.68	-	-	-	-	0.003	0.093
3	1.78	0.10	-	-	-	-	0.009	0.062
4 (Ref)	0.92	0.03	0.11	-	-	-	0.005	0.043
5 (Ref)	0.25	0.06	-	-	-	-	0.003	0.039
6	-	-	-	1.50	0.50	1.75	0.002	0.092

Table 1 shows the chemical composition of steel powder according to the invention and reference materials.

The obtained steel powders 1-5 were mixed with 0.5 % and 0.7 % by weight of the composition, respectively, of graphite UF4, available from Kropfmühle, Germany and 0.8 % of Amide wax PM, available from Höganäs AB, Sweden.

Powder 4 was outside the boundaries of the present invention being alloyed with 0.11 wt% vanadium and having a manganese content of 0.03 wt%. Powder 5 had both manganese content and chromium content below the boundaries of the present invention.

A reference mix, based on DISTALOY AB (powder 6) was also prepared. In this case the composition prepared contained 0.5 % of graphite and 0.8 % of Amide Wax PM.

The obtained powder compositions were transferred to a die and compacted to form tensile tests bars at a compaction pressure of 600 MPa. The compacted tests bars were further sintered in a laboratory belt furnace at 1120 °C for 30 minutes in an atmosphere of 90 % nitrogen and 10 % of hydrogen.

The sintered samples were tested with respect to tensile strength and elongation according to ASTME9-89C and hardness, HV10 according to EN ISO 6507-1. The samples were also analysed with respect to the carbon and oxygen content.

Impact energy was tested in accordance with EN10045-1.

The following Table 2 shows added amount of graphite, results from chemical analysis, and results from tensile and hardness testing.

Table 2

Powder Composition based on powder	Added Graphite [%]	C [%]	O [%]	Yield strength [MPa]	Tensile strength [MPa]	Elongation [%]	Hardness, HV10	IE [J]
1	0.5	0.50	0.05	345	512	4.6	139	25
1	0.7	0.69	0.05	417	627	3.0	178	23
2	0,5	0.55	0.07	371	540	3.3	168	24
2	0.7	0.72	0.07	398	611	3.0	181	21
3	0,5	0.56	0.03	427	625	3.1	172	28
3	0.7	0.72	0.03	496	697	2.3	195	23
4 (Ref)	0.5	0.51	0.03	379	517	2.8	156	19
4 (Ref)	0.7	0.70	0.03	462	610	1.6	192	15
5 (Ref)	0.5	0.48	0.02	287	391	4.2	113	23
5 (Ref)	0.7	0.67	0.02	331	478	3.2	143	19
6- Dist AB	0.5	0.48	0.01	363	610	2.8	178	28

Table 2 shows the amount of added graphite to the compositions, analysed C and O content of the produced samples, as well as results from tensile test and hardness testing of the produced samples.

At 0.7 % added graphite, samples based on powder 1, 2 showed comparable or better values than DISTALOY AB mixed with 0.5 % graphite powder for yield strength, tensile strength, elongation, and hardness. Impact energy was slightly below but still sufficiently good, slightly better for powder 1 than for powder 2.

Already at 0.5 % added graphite, samples based on powder 3 showed comparable or better values than DISTALOY AB mixed with 0.5 % graphite powder for yield strength, tensile strength, elongation. Also impact energy and hardness matches DISTALOY AB.

For samples based on powder 4 elongation and impact energy are much lower than the values of DISTALOY AB at comparable tensile strength. For samples based on powder 5 it can be seen that impact energy and elongation are decreasing with increasing carbon content and would be much lower if even higher graphite additions would be applied to increase tensile strength to a comparable level with DISTALOY AB.

CLAIMS

1. A water atomised prealloyed iron-based steel powder which comprises by weight-%:
   0.4-2.0 Cr,
   0.1-0.8 Mn,
   less than 0.1 V,
   less than 0.1 Mo,
   less than 0.1 Ni,
   less than 0.2 Cu,
   less than 0.1 C,
   less than 0.25 O,
   less than 0.5 of unavoidable impurities, and
   the balance being iron.

2. A powder according to claim 1, wherein the content of Cr is at most 1.8 weight-%, preferably at most 1.5 weight-%.

3. A powder according to claim 1 or 2, wherein the content of Cr is at least 0.5 weight-%.

4. A powder according to anyone of claims 1-3, wherein the content of Mn is at least 0.2 weight-%, preferably at least 0.3 weight-%.

5. A powder according to anyone of claims 1-4, wherein the content of Mn is at most 0.7 weight-%, preferably at most 0.6 weight-%.

6. A powder according to anyone of claims 1-5, wherein the sum of the chromium and manganese content is less than 2.5 wt%, preferably less than 2.3 wt% and most preferably less than 1.9 % by weight.

7. A powder according to anyone of claims 1, wherein the content of Cr is 0.4-0.6 weight-%, and the content of Mn is 0.6-0.8 weight-%, preferably the content of Mn is 0.7-0.8 weight-%.

8. A powder according to anyone of claims 1, wherein the content of Cr is at least 0.7 weight-%, and the content of Mn is at most 0.5 weight-%.

9. A powder according to anyone of claims 1, wherein the content of Cr is at least 1.0 weight-%, and the content of Mn is at most 0.4 weight-%, preferably at most 0.3 weight-%.

10. An iron-based powder composition comprising a steel powder according to any of claim 1-9 mixed with 0.35-1 % by weight of the composition of graphite, 0.05-2 % by weight of the composition of lubricants and optionally copper in an amount up to 3 %, and optionally hard phase materials and machinability enhancing agents.

11. A method of producing a sintered component comprising the steps of:
a) preparing an iron-based steel powder mixture having the composition claimed in claim 10,
b) subjecting the composition to compaction between 400 and 2000 MPa,
c) sintering the obtained green component in a reducing atmosphere at temperature between 1 000-1 400 ^{o}C,
d) optionally forging the heated component at a temperature above 500 ^{o}C or subjecting the obtained sintered component to a heat treatment or hardening step.

12. A sintered component produced from the powder composition according to claim 10 having a pearlitic/ferritic microstructure.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2008/051511

A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: B22F, C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 58130249 A, SUMITOMO METAL IND LTD, 1983-08-03: (abstract) Retrieved from PAJ database, table 1 --	1-12
Y	US 6027544 A (ARVIDSSON J.), 22 February 2000 (22.02.2000), claims 1-2, abstract --	1-12
A	US 4266974 A (NITTA M. ET AL), 12 May 1981 (12.05.1981), abstract --	1-12
A	JP 59035602 A, SUMITOMO METAL IND LTD, 1984-02-27: (abstract) Retrieved from: PAJ database --	1-12

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
8 April 2009	09-04-2009

Name and mailing address of the ISA/	Authorized officer
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C22C 33/02 (2006.01)
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Cited literature, if any, will be enclosed in paper form.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2008/051511

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 01025901 A, KOBE STEEL LTD, 1989-01-27: (abstract) Retrieved from: PAJ database -- --------	1-12

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/SE2008/051511

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