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(71) Applicant: **Höganäs AB (publ)**
263 83 Höganäs (SE)

(72) Inventor: **Larsson, Anna**
26060 Kvidinge (SE)

(54) **IRON BASED POWDERS FOR POWDER INJECTION MOLDING**

(57) The present invention relates to a feedstock for metal injection molding, comprising a coarse stainless steel powder, having an median particle size of 20-60μm, and 99% of the particles less than 120 μm, wherein the iron-based powder comprises, by weight percent;15-17%Cr; 3-5% Ni; 3-5%,Cu; 0.15-0.45% Nb; <1.0% Mn; <1.0% Si; less than 0.08% C; and a binder.

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Description

FIELD OF THE INVENTION

[0001] The present invention concerns an iron-based powder, in particular a stainless steel powder, which is useful for powder injection molding; a composition for powder injection molding; a method of making sintered components from the powder composition; and sintered components made from the powder composition. Using the powder composition it may be possible to obtain sintered parts with densities above 96% of the theoretical density, thus resulting in excellent mechanical properties.

BACKGROUND OF THE INVENTION

[0002] Powder injection molding, also called metal injection molding (MIM) is an interesting technique for producing high density sintered components of complex shapes. In general, fine carbonyl iron powders are used in this process. Other types of powders used are gas-atomized or water-atomized of very fine particle size, the cost of which is relatively high. In order to improve the competitiveness of the MIM process it is desirable to reduce the cost of the powder used. One way of achieving this is by utilizing coarser powders. However, coarse powders have a lower surface energy than fine powders and are thus much less active during sintering. Another issue is that the use of coarse and irregular powders leads a lower packing density and thus the maximal powder content of the feedstock is limited. Lower powder content results in a higher shrinkage during sintering and may lead to, *inter alia,* high dimensional scatter between components produced in a production run. WO2012089807 discloses the use of a coarse powder which achieves a theoretical density of more than 95%. There is still a need for technology which can achieve even higher density.

[0003] Normally, the solid loading (*i.e.* the portion of iron- based powder) of an iron-based MIM feedstock (*i.e.* the iron-based powder mixed with organic binder ready to be injected) is about 50% by volume, which means that, in order to reach high density after sintering (above 93% of theoretical density), the green component must shrink almost by 50% by volume. This is in contrast to PM components produced through uniaxial compaction which already in green state obtain relatively high density. Therefore, fine powders having high sintering activity are normally used in MIM. By elevating the sintering temperature coarser powders may be used. This, however, results in grain coarsening which in turn gives mechanical properties which are not optimal.

[0004] It has unexpectedly been found that a coarse metal powder, wherein the metal powder has a certain composition, can be used in a feedstock for powder injection molding in order to obtain components with a sintered density of at least 96% of the theoretical density.

SUMMARY

[0005] One object of the invention is to provide a relatively coarse stainless steel powder composition having low amounts of alloying elements which is suitable for metal injection molding.

[0006] Another object of invention is to provide a metal injection molding feedstock composition comprising said relatively coarse stainless steel powder composition.

[0007] Another object of the invention is to provide a method for producing injection molded sintered components from the feedstock composition, said components having a density of at least 96% of the theoretical density.

[0008] Still another object of the present invention is to provide a sintered component produced according to the MIM process having a density of 96% and above, of theoretical density and a tensile strength above 800MPa as sintered, without hardening.

[0009] At least one of these objects is accomplished by:

An iron based powder composition for metal injection moulding having an median particle size of 20-60μm, preferably 20-45μm, most preferably 25-45μm, or even more preferred 25-35μm. The particle size is determined by laser diffraction using a Sympatec Helos instrument. The median particle size as defined above means that 50% of the particles in the powder is larger than this value. This value is normally termed the "X50" value.

[0010] A metal injection molding feedstock composition comprising atomized iron-based powder composition with an median particle size of 20-60μm, preferably 20-45μm, most preferably 25-45μm, or even more preferred 25-35μm, and an organic binder.

[0011] A method for producing a sintered component comprising the steps of:

a) preparing a metal injection molding feedstock as suggested above;
b) molding the feedstock into an unsintered blank;

c) removing the organic binder;
d) sintering the obtained blank in a reducing atmosphere at a temperature between 1 200-1 400° C.
e) cooling the sintered component, and;
f) optionally subjecting the component to post sintering treatment such as precipitation hardening, case hardening, nitriding, carburizing,

nitrocarburizing, carbonitriding, induction hardening, surface rolling and/or shot peening.

[0012] A sintered component made from the feedstock composition, the component having a density of at least 96% of theoretical density, and a tensile strength above 800MPa.

DETAILED DESCRIPTION OF THE INVENTION

[0013] The stainless steel powder composition includes at least one iron based powder and/or pure iron powder. The iron based powder and/or pure iron powder can be produced by water or gas atomization of an iron melt and optionally alloying elements. The atomized powder can further be subjected to a reduction annealing process, and optionally be furthered alloyed by using a diffusion alloying process. Alternatively, iron powder may be produced by reduction of iron-oxides.

[0014] The particle size of the iron- or iron- based powder composition is such that the median particle size is of 20-60μm, preferably 20-45μm, most preferably 25-45 μm, or even more preferably 25-35μm. Further, it is preferred that X_{99} shall be at most 120μm, preferably at most 100 μm. (X_{99} means that 99% of the particles have a particle size less than X_{99})

[0015] Copper, Cu will enhance the strength and hardness through solid solution hardening. Cu, 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. The powder may optionally be admixed with Cu, preferably in the form of Cu-powder in an amount of 0-5-wt%, or 3-5-wt%. Other substances such as hard phase materials and machinability enhancing agents, such as MnS, MoS_2, CaF_2, different kinds of minerals etc. may optionally be added to the iron based powder composition.

[0016] The feedstock composition may be prepared by mixing the iron based powder composition described above and a binder.

[0017] The binder in the form of at least one organic binder may be present in the feedstock composition in a concentration of 30-65% by volume, preferably 35-60% by volume, more preferably 40-55% by volume. When using the term binder in the present description also other organic substances that are commonly in MIM-feedstocks are included, such as e.g. releasing agents, lubricants, wetting agents, rheology modifiers, dispersant agents. Examples of suitable organic binders are waxes, polyolefins, such as polyethylenes and polypropylenes, polystyrenes, polyvinyl chloride, polyethylene carbonate, polyethylene glycol, stearic acids and polyoxymethylen.

[0018] The feedstock composition is molded into a blank. The obtained blank is then heat treated, or treated in a solvent or by other means to remove one part of the binder as is known in the art, and then further subjected to sintering in a reducing atmosphere in vacuum or in reduced pressure, at a temperature of about 1200-1400°C.

[0019] The sintered component may be subjected to a heat treatment process in order to obtain a desired microstructure, *e.g.* by heat treatment and by controlled cooling rate. The hardening process may include known processes such as precipitation hardening, quench and temper, case hardening, nitriding, carburizing, nitrocarburizing, carbonitriding, induction hardening and the like. Alternatively, a sinter-hardening process at high cooling rate may be utilized. Other types of post sintering treatments may be utilized, such as surface rolling or shot peening which introduce compressive residual stresses enhancing the fatigue life.

[0020] Sintered components according to the invention reach a sintered density of at least 96% of the theoretical density, and tensile strength above 800 MPa.

EXAMPLE 1

[0021] Iron based powder compositions according to Table 1 were prepared.

Table 1

Element	A	B	D	E	C (comparative)
Cr	16.5	16.5	17	16.5	16.1
Ni	4.09	4.3	4.3	4.09	13.3
Cu	4	4.04	3.96	4	

(continued)

Element	A	B	D	E	C (comparative)
Nb	0.37	0.37	0.47	0.37	
Mn	0.1	0.1	0.04	0.1	0.096
Si	0.68	0.53	0.95	0.68	0.881
Mo					2.12
C	0.016	0.079	0.011	0.016	0.022
O	0.351	0.433	0.146	0.351	0.236
N	0.04	0.025	0.021	0.04	0.044
S	0.007	0.006	0.003	0.007	0.009
Fe	Bal	Bal	Bal	Bal	Bal
X10	10.9	14.2	14.4	21.4	12.2
X50	24.4	32.6	31.0	35.0	26.4
X90	46.7	57.0	52.1	56.7	46.9
x99	72.2	79.8	86.8	104.0	66.9

Example 2

[0022] The compositions were compacted to a density about 4.5g/cm^3 (58% of theoretical density) into cylinders with a diameter 25mm and a height of 8mm and thereafter A, C and E were sintered at 1350°C in an atmosphere of 100%H_2 by volume, during 1200 minutes. Sample C was sintered at 1380°C, during 120 minutes, 100% H_2. Sintered density was measured using the water displacement method as described in standard SS-EN ISO 3369:2010.

Table 2 shows the test results.

	A	C (comparative)	E
SD	**7.63**	**6.65**	**7.37**
% of theoretical density	**98.2**	**83.4**	**95.0**

Example 3

[0023] A feedstock containing the metal powder composition A, B, and D, respectively, were prepared and compared with a feedstock made from composition C, by mixing the powder compositions with an organic binder. The binder was composed of 47.5 % polyethylene, 47.5% paraffin wax and 5% stearic acid. All percentage in weight percentage. The organic binder and the powder compositions were mixed in a ratio of the metal powder:binder of 53:47 by volume.

[0024] The feedstock were injection molded into standard MIM tensile bars according to ISO- SS EN ISO 2740 The samples were then debound in hexane for 4 hours at 60°C to remove the paraffin wax, followed by sintering at 1350°C in an atmosphere of 100% hydrogen for 120 minutes.

[0025] The Sintered Density were measured using the water displacement method. Tensile test was tested according to SS EN ISO 2740. Results are shown in table 3. Standard values were taken from ISO22068 and shows values for the standard alloys 17-4PH and 316L in the sintered state. The mechanical properties are presented as % of standard value in order to be able to compare two different alloys.

Table 3

	A		B		D		C	
	Absolute value	% of standard value	Absolute value	% of standard value	Absolute value	% of standard value	Absolute value	% of standard value
Sintered Density (g/cm^3)	7,68		7,68		7,69		7,38	
Hardness (HRC)	27.8	103	26.4	98	29,8	110	58.2	49
Tensile strength (MPa)	1129	141	1124	141	1086	135	286.5	64
Yield strength 0.2% (MPa)	897	138	877	135	860	132	130.9	94
Elongation (%)	2.9	97	2.84	95	1,4	47	22.81	57

Claims

1. Feedstock for metal injection molding, comprising;

a) an iron based powder, having an median particle size of 20-60μm, and 99% of the particles less than 120 μm, wherein the iron-based powder comprises, by weight percent;
15-17%Cr; 3-5% Ni; 3-5%,Cu; 0.15-0.45% Nb; <1.0% Mn; <1.0% Si; less than 0.08% C; and
b) a binder.

2. Use of a feedstock according to claim 1 for metal injection molding.

3. Use according to claim 2, comprising the steps of:

a) preparing a metal injection molding feedstock according to claim 1,
b) molding the feedstock into an unsintered blank,
c) removing the organic binder
d) sintering the obtained blank in a reducing atmosphere at a temperature between 1 200-1 400°C
e) cooling the sintered component, and
f) optionally subjecting the component to post sintering treatment such as precipitation hardening, case hardening, nitriding, carburizing, nitrocarburizing, carbonitriding, induction hardening, surface rolling and/or shot peening.

4. A sintered component produced according to claim 3 having a density of at least 96% of the theoretical density.

5. A sintered component according to claim 4 having a tensile strength above 800MPa.

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EUROPEAN SEARCH REPORT

Application Number
EP 15 18 9896

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	JOYS J ET AL: "Design of experiments (DOE) study to achieve higher mechanical properties by optimizing particle size distribution and processing parameters of 17-4PH Stainless Steel powder for Metal Injection Molding (MIM).", ADVANCES IN POWDER METALLURGY AND PARTICULATE MATERIALS, 2013, pages FP, 01-9-01-16, XP008179964, Proceedings of the 2013 international Conference on Powder Metallurgy & Particulate Materials sponsored by the Metal Powder Industries Federation * Experiment; paragraph [0002] * * tables 1, 7 * * figure 6 * -----	1-5	INV. B22F3/22 C22C33/02 C22C38/42 B22F1/00
X	MURRAY K ET AL: "Effect of particle size distribution on processing and properties of MIM 17-4PH", ADVANCES IN POWDER METALLURGY AND PARTICULATE MATERIALS - 2010, PROCEEDINGS OF THE 2010 INTERNATIONAL CONFERENCE ON POWDER METALLURGY AND PARTICULATE MATERIALS, POWDERMET 2010 - ADVANCES IN POWDER METALLURGY AND PARTICULATE MATERIALS - 2010, PROCEEDI, 2010, XP002756955,	4,5	TECHNICAL FIELDS SEARCHED (IPC)
Y	* Experimental Procedure * * figures 6, 11, 12 * * Summary and conclusions * -----	1-3	B22F C22C
Y,D	WO 2012/089807 A1 (HOEGANAES AB PUBL [SE]; LARSSON ANNA [SE]) 5 July 2012 (2012-07-05)	1-3	
A	* page 2, line 25 - page 3, line 2 * * page 5, line 9 - line 13 * * table 4 * -----	4,5	

The present search report has been drawn up for all claims

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Place of search	Date of completion of the search	Examiner
The Hague	22 April 2016	Morra, Valentina

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 15 18 9896

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22-04-2016

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

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