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(54) METAL POWDER

METALLPULVER

POUDRE MÉTALLIQUE

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(56) References cited:
US-A1- 2001 027 170
US-A1- 2010 116 240
US-A1- 2012 187 611
US-B2- 7 993 429

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Description

Field of the Invention

[0001] The present invention relates to a new metal powder composition for the powder metallurgical industry. Particularly the invention relates to a sponge-iron-based powder composition which includes a lubricant for improving powder properties, compaction and processing.

Background of the Invention

[0002] In industry, the use of metal products manufactured by compacting and sintering iron-based powder compositions is becoming increasingly widespread. The quality requirements of these metal products are continuously raised and, as a consequence, new powder compositions having improved properties need to be developed.

[0003] There is, thus, a need to improve powder properties, the compaction process and also the sinter process. Parameters that may be improved are e.g. characteristics of the metal powder itself, or the type of, or characteristics, of various additives. Additives may include alloying elements, flow agents, lubricants, machinability enhancing agents, or hard phase materials. As lubricants for low density PM applications, zinc stearate or amide wax are commonly used due to their overall good performance.

[0004] As regards characteristics of the metal powder itself, interest has focussed on the use of atomised metal powder, and in particular on the use thereof together with certain types of lubricant. Atomized metal powder may be prepared by disintegration of a thin stream of molten metal through the impingement of high energy jets of a fluid (e.g. water). Atomized metal powder may be advantageous if high green density is sought in powder metallurgy structural parts.

[0005] A first example of the use of atomized metal powder together with particular lubricants is in US 2012/0187611 which reports the use of atomized metal powder together with a lubricating combination of three components, named substances A, B and C. Polyethylene wax is favoured for substance A. Options for substance B include fatty acid amides, fatty acid bisamides, saturated fatty alcohols and saturated fatty acid glycerols. Substance C is an amide oligomer which may have a molecular weight of between 500 and 30,000. Substance C is generally used as the main component of the lubricant. Substance A is reported to have a negative effect on ejection behaviour. Substance B is used in an amount which is at least half as much as the amount of substance A, in order to compensate for this negative effect seen in the exemplified metal powder compositions, which are all based on atomised metal powders.

[0006] A second example is in US 2001/0027170 which reports the use of atomized metal powder together with a lubricant combination of aggregate particles having a core of a first lubricant (ethylene bis-stearamide (EBS) is preferred), the surface of the core being coated with particles of a second lubricant (preferably zinc stearate). The Examples report that this particular arrangement (i.e. having particles of the second lubricant located on the surface of core particles of the first lubricant) enables improved flow for resulting compositions based on atomised metal powder, as compared to other ways of combining the first and second lubricants.

[0007] A third example is in US 7,993,429 which reports the use of atomized metal powder together with composite lubricant particles having a core comprising a solid organic lubricant, with fine carbon particles adhered to the surface. Preferred solid organic lubricants for the core include fatty acids, fatty acid monoamides and fatty acid bisamides. The Examples report that having the carbon particles adhered to the surface of the solid organic cores helps avoid agglomeration and improve flow for resulting compositions based on atomised metal powder.

[0008] Another type of metal powder is the so-called sponge iron powder. Components which are made by compacting sponge iron powder, have a green strength which is quite high compared to the green strength obtained when compacting e.g. atomized powder. In order to improve performance during compaction and ejection of compacted components from the tools, a lubricant is normally added to the powder mixture. This has the drawback of e.g. reducing the flow rate of the powder which may cause longer filling time.

[0009] In particular, the sponge powders exhibit a lower flow rate compared to that of atomized powder. This further complicates the use of lubricants in sponge iron based powder compositions.

[0010] Adding commonly used lubricants, such as metal stearate, to metal powder may result in residual lubricant deposits in the furnaces used for sintering, and also results in surface defects in the final product, leading to higher scrap rates and costly maintenance.

[0011] As regards examples of uses of lubricants with sponge metal powders, reference may be made to e.g. GB 391,155 wherein a fatty acid such as stearic acid, palmitic acid or oleic acid may be used to lubricant sponge iron. A more recent example is US2010/0116240, where a synthetic wax such as ethylene bis-stearamide wax is described for use with sponge iron powder.

Summary of the Invention

[0012] The present invention is based, inter alia, on the surprising finding that for sponge iron particles (or sponge iron-based particles), the use of a combination of behenamide, stearamide and palmitamide helps address the flowability issue noted above. In particular, the use of such combinations enables the provision of metal powder compositions having excellent flowability, whilst also providing a suitable apparent density. The use of such combinations also provides further advantages which will become apparent from the description of the invention as set out below.

[0013] Thus, the present invention relates to a metal powder composition comprising sponge iron particles (or sponge iron-based particles) and a lubricant according to claim 1. The lubricant imparts lower ejection force in the process of manufacturing compacted components, with a minimal negative influence on the flow rate.

Detailed Description of the Invention

[0014] Sponge iron is produced from direct reduction of iron ore (in the form of lumps, pellets or fines) by a reducing gas which may be produced from natural gas or coal. Such iron can be milled or crushed to produce particles. These particles typically have an irregular shape, high surface area, and internal porosity. A plurality of such particles forms a powder. The metal powder may be annealed after milling or crushing.

[0015] The present invention provides a metal powder composition comprising (i) sponge iron particles or sponge iron-based particles, and (ii) a lubricant comprising a mixture of behenamide, stearamide and palmitamide, wherein the amount of lubricant is between 0.2wt% and 1.4wt%. The sponge iron powder particles may consist essentially of iron or may be so-called iron-based and include other alloying elements, such as C, Cu, Ni, or Mo (preferably Cu, Ni, or Mo; alternatively, in one particular preferred embodiment, both C and Cu are used). When C is used as an alloying element, it is preferably used in the form of graphite.

[0016] Thus, in one preferred aspect of the invention component (i) of the metal powder composition is sponge iron-based particles which comprise sponge iron particles together with one or more of C, Cu, Ni and Mo (as alloying elements). In particular, the sponge iron-based particles preferably comprise sponge iron particles together with particles of one or more of C, Cu, Ni and Mo. In both of these aspects it is preferred for both C and Cu to be used.

[0017] Preferably in this regard the sponge iron-based particles comprise at least 80% by weight, more preferably at least 90% by weight, and more preferably still at least 95% by weight of sponge iron particles. Thus, the sponge iron-based particles preferably comprise 20% by weight or less, more preferably 10% by weight or less, and more preferably still 5% by weight or less of the alloying element(s) (which, as noted above, are preferably one or more of C, Cu, Ni and Mo, typically in particulate form).

[0018] The metal powder compositions according to the invention contain sponge iron- or sponge iron-based powders, such as MH80.23, NC100.24 and SC100.26 (available from Höganäs AB, Sweden), optionally at least one alloying element, and at least one lubricant.

[0019] Alloying elements which can be added in powder form to the iron powder may include graphite, or metal powders other than iron (such as Cu, Ni, or Mo).

[0020] In a preferred embodiment, the lubricant for use in the present invention (and preferably also the metal powder composition of the present invention) is essentially free of organic lubricants other than the fatty acid amides. In particular, it is preferably essentially free of organic lubricants other than the preferred fatty acid monoamides for use in the present invention as described herein. Thus, it is preferably essentially free of fatty acids, fatty acid bisamides, fatty acid alcohols, fatty acid glycerols, and/or relatively heavy amides (e.g. amide oligomers with a molecular weight of 500 g/mol or more). It is also preferably essentially free of metal soaps, such as zinc stearate. The avoidance of metal-containing lubricants such as metal soaps is advantageous because it reduces the amount of undesirable "ash" residue after the lubricant has decomposed.

[0021] In invention, the lubricant is a mixture of behenamide, stearamide and palmitamide. The amounts are preferably 20-50wt% stearamide, 20-50wt% palmitamide, and may further include arachidamide, the balance being behenamide.

[0022] More preferably, the amounts are 25wt% stearamide, 25wt% palmitamide, and 50wt% behenamide.

[0023] The present disclosure relates to a metal powder composition comprising sponge iron particles and a lubricant. Preferably, the lubricant is a mixture of behenamid, stearamide and palmitamide.

[0024] The lubricant is preferably in particulate form. In this regard, the lubricant may comprise separate particles of each of the said at least two different fatty acid amides. Alternatively, the lubricant may be a mixture of the different fatty acid amides in particulate form (i.e. with each individual particle generally comprising a mixture of the different fatty acid amides).

[0025] The lubricant amount is between 0.2wt% and 1.4wt%, preferably between 0.4wt% and 1.0wt%, or more preferably between 0.6wt% and 1.0wt%. Thus, the metal powder composition of the present invention comprises the lubricant in such amounts.

[0026] Working temperature of the lubricant ranges from RT (room temperature) in the compaction tool and to standard working temperatures in longer series in production, e.g. ~60°C and up to 80°C.

[0027] In the metal powder composition of the present invention, the sponge iron particles preferably have an average particle size of at least 5 μm, more preferably at least 10 μm, more preferably still at least 20 μm, and more preferably still at least 50 μm. The sponge iron particles preferably have an average particle size of 500 μm or less, more preferably 300 μm or less, more preferably still 200 μm or less, and more preferably still 150 μm or less.

[0028] Alloying elements, when used, are preferably used in particulate form. In this regard the alloying element particles preferably have an average particle size of at least 5 μm, more preferably at least 10 μm, more preferably still at least 20 μm, and more preferably still at least 50 μm. The alloying element particles preferably have an average particle size of 500 μm or less, more preferably 300 μm or less, more preferably still 200 μm or less, and more preferably still 150 μm or less.

[0029] As noted above, the lubricant may preferably be used in particulate form. In this regard the lubricant particles preferably have an average particle size of at least 0.5 μm, more preferably at least 1 μm, more preferably still at least 2 μm, and more preferably still at least 5 μm. The lubricant particles preferably have an average particle size of 500 μm or less, more preferably 200 μm or less, more preferably still 100 μm or less, and more preferably still 50 μm or less.

[0030] As used herein, average particle size preferably refers to the average particle size as measured by a laser diffraction scattering method.

[0031] The powder composition has better flow which increases productivity and quality of the final component.

[0032] The powder system exhibits low friction during the compaction operation and reduces the ejection forces and ejection energies that occur during ejection of the component from the compaction tool. A reduction of these energies results in less tool wear and less surface defects in the final product.

[0033] The green strength is also improved and this mitigates the risk for green cracks and other "green" related damages on components before the sintering operation. Higher green strength improves production efficiency and reduces scrap rates in production.

[0034] By using the new mixed powder system it is possible to reduce or avoid surface defects that normally appear when using conventional lubricants in metal powder mixes.

[0035] The present invention also provides a process comprising (i) compacting a metal powder composition of the present invention as defined above, and (ii) sintering the thus obtained compacted metal powder composition to produce a metal product. The present invention also provides a metal product obtainable or obtained by such a process.

Example 1

[0036] Different iron-based powder metallurgical mixtures, according to Table 1, were prepared. As iron-based powder the sponge iron powders MH80.23, NC100.24 and SC100.26 (available from Höganäs AB, Sweden), were used. Also used were ABC100.30 (atomized iron powder), 2%Cu-100mesh (copper powder from Poemton, Italy), DACu (Distaloy ACu available from Höganäs AB, Sweden) and 0,5%C-UF4 (graphite from KropfmÜhl AG, Germany).

Table 1.

Mark	Chemistry	Supplier
X	50%Behenamide/25%stearamide/25%palmitamide	Abril industrial waxes
Znst	Zinc stearate	Faci, UK
Amidewax PM	Ethylene bisstearamide	Faci, Italy
PS	50%Palmite/50%Stearamide	Croda, UK
B	17%Behenamide/46%arachidic amide/37%stearamide	

Table 2. Iron-based powder metallurgical mixtures prepared

1(inv)	NCx0.4	NC100.24+2%Cu-100+0.5%C-UF4+0,4%X
2(inv)	NCx0.6	NC100.24+2%Cu-100+0.5%C-UF4+0,6%X
3(inv)	NCx0.8	NC100.24+2%Cu-100+0.5%C-UF4+0,8%X
4(inv)	NCx1.0	NC100.24+2%Cu-100+0.5°sC-UF4+1,0%X
5(comp)	NCZ0.8	NC100.24+2%Cu-100+0.5%C-UF4+0,8%ZnSt
6(comp)	NCA0.8	NC100.24+2%Cu-100+0.5%C-UF4+0,8%AmideWax PM

(continued)

7(inv)	MHx0.8	MH80.23+2%Cu-100+0.5%C-UF4+0.8%X
8(comp)	MHZ0.8	MH80.23+2%Cu-100+0.5%C-UF4+0.8%ZnSt
9(comp)	MHA0.8	MH80.23+2%Cu-100+0.5%C-UF4+0.8%AmideWax PM
10(inv)	NCB0.8	NC100.24+2%Cu-100+0.5%C-UF4+0,8%B
11(comp)	ABCx0.8	ABC100.30+1,5%Cu (DACu)+0.5%C-UF4+0.8%X
12(comp)	ABCA0.8	ABC100.30+1,5%Cu (DACu)+0.5%C-UF4+0.8% Amide Wax
		PM
13(comp)	ABCPS0.8	ABC100.30+1,5%Cu (DACu)+0.5%C-UF4+0.8%PS

[0037] The Hall flow (FH) rate was measured according to ISO 4490 Flow Gustavsson (FG) and according to ISO13517:2013 and the apparent density was measured according to ISO 3923.

Table 3. Flow rate (FH and FG) and Apparent density (AD) of iron-based powder metallurgical mixtures

Mix		AD (g/cm³)	FH (s/50g)	FG (s/50g)
1(inv)	NCx0.4	2,49	32	36
2(inv)	Nix0.6	2,49	33	36
3(inv)	NCx0.8	2,5	33	37
4(inv)	Nix1.0	2,5	34	38
5(comp)	NCZ0.8	2,72	35	41
6(comp)	NCA0.8	2,5	39	45
7(inv)	MHx0.8	2,34	34	35
8(comp)	MHZ0.8	2,45	35	37
9(comp)	MHA0.8	2,32	40	41
10(inv)	NCB0.8	2,47	37	42
11(comp)	ABCx0.8	3,06	29	
12(comp)	ABCA0.8	3,04	30	
13(comp)	ABCPS0.8	3,07	29	

[0038] Table 3 shows that the new sponge iron powder mix shows similar apparent density levels as for mixes with amide wax and highest apparent density was obtained for mixes with zinc stearate. All mixes with X show improved flowability according to the two different methods to measure flow. Also, the mix with -B (Mix 10) improved flowability as compared to the metal-free bisamide wax (Mix 6).

Table 4. Flow rate (FH) and apparent AD (AD) of the metal powders, without lubricant.

Iron powder	AD (g/cm³)	FH (Sec/50g)
NC100.24	2,43	32
SC100.26	2,65	30
MH80.23	2,30	34
ABC100.30	2,99	23

[0039] For all mixes, the lubricating properties were measured, by recording the total energy per enveloped area needed in order to eject a compacted sample from the die as well as the peak ejection force per enveloped area. The components were cylindrical having a diameter of 25 mm, and a height of 15 mm, and the compaction pressures applied were 250, 400 and 550MPa.

Table 5 Peak ejection force and ejection energy

Mix		EE250 (J/cm^2)	EE400 (J/cm^2)	EE550 (J/cm^2)	EF250 (N/mm^2)	EF400 (N/mm^2)	EF550 (N/mm^2)
1 (inv)	NCx0.4	18,7	31,6	43,4	23,3	25,9	34,3
2 (inv)	Nix0.6	19	30,2	40,8	19,5	22,7	32
3 (inv)	NCx0.8	18,7	29,6	37,7	18,6	24,3	32,3
4 (inv)	Nix1.0	19,9	28,5	36,5	20,8	23	29,2
5 (comp)	NCZ0.8	21,9	33,2	47	23,5	25,8	35,5
6 (comp)	NCA0.8	22	34	42,5	22,5	25,4	34,5
7 (inv)	MHx0.8	17,6	27,8	37,9	21,2	23, 8	29, 6
8 (comp)	MHZ0.8	20,6	32,2	45,7	22,3	26	32
9 (comp)	MHA0.8	20,9	31,4	42,5	23	24	31,9
10 (inv)	NCB0.8	21,1	33,0	42,6	18,3	22,5	29,4
11 (comp)	ABCx0.8	24,6	33,3	38,9	16,1	22,7	28,1
12 (comp)	ABCA0.8	25,7	33,3	38	17,2	23,8	27,2
13 (comp)	ABCPS0.8	26,6	34,6	39,9	17	22,8	27

[0040] Table 5 for mixes 1 to 6 shows that reduced amounts of X with NC100.24 give similar properties as for zinc stearate and amide wax mixes at 0,8% lubricant levels. The use of B (in Mix 10) also gave similar results. Overall, X scored better than the comparative examples except for the static ejection forces at 550MPa with SC100.26 as base powder.

[0041] Green strength at 6.45g/cm^3 was measured on all prepared mixes. The green strength was tested on a TRS test bar.

Table 6. Green strength

Mix		GS at 6.45g/cm^3 (N/mm^2)
1	NCx0.4	24
2	Nix0.6	24
3	NCx0.8	23
4	Nix1.0	23
5	NCZ0.8	12
6	NCA0.8	15
7	MHx0.8	31
8	MHZ0.8	21
9	MHA0.8	24
10	NCB0.8	19

[0042] Green strength comparison for NC100.24 mixes shows improvements with 50 to 75% when using X and improvements were also seen when using B. For mixes with MH80.23 the green strength increase were 30 to 50% better with X.

Claims

1. Metal powder composition comprising (i) sponge iron particles or sponge iron-based particles, and (ii) a lubricant comprising a mixture of behenamide, stearamide and palmitamide wherein the amount of lubricant is between 0.2wt% and 1.4wt%.

2. Metal powder composition according to claim 1, wherein component (i) of the metal powder composition is sponge iron-based particles which comprise sponge iron particles together with one or more of C, Cu, Ni and Mo.

Patentansprüche

1. Metallpulverzusammensetzung, umfassend (i) Eisenschwammpartikel oder Partikel auf Eisenschwammbasis und (ii) ein Schmiermittel, das eine Mischung aus Behenamid, Stearamid und Palmitamid umfasst, wobei die Menge an Schmiermittel zwischen 0,2 Gew.-% und 1,4 Gew.-% beträgt.

2. Metallpulverzusammensetzung nach Anspruch 1, wobei es sich bei Komponente (i) der Metallpulverzusammensetzung um Partikel auf Eisenschwammbasis handelt, die zusammen mit C, Cu, Ni und/oder Mo Eisenschwammpartikel umfassen.

Revendications

1. Composition de poudre métallique comprenant (i) des particules de fer spongieux ou des particules à base de fer spongieux, et (ii) un lubrifiant comprenant un mélange de béhénamide, de stéaramide et de palmitamide, la quantité de lubrifiant étant comprise entre 0,2 % en poids et 1,4 % en poids.

2. Composition de poudre métallique selon la revendication 1, dans laquelle un composant (i) de la composition de poudre métallique est des particules à base de fer spongieux qui comprennent des particules de fer spongieux conjointement avec un ou plusieurs éléments parmi C, Cu, Ni et Mo.

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- US 20120187611 A **[0005]**
- US 20010027170 A **[0006]**
- US 7993429 B **[0007]**
- GB 391155 A **[0011]**
- US 20100116240 A **[0011]**