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(54) **Dry powder metal compositions and methods of making and using the same**

(57) The present invention provides powder metal compositions and methods of making and using the same. Powder metal compositions according to the invention are substantially dry and include a blend of base metal particles and a pressing aid. The pressing aid includes a micronized deformable solid material and a high melt point lubricating material, which have been pre-

mixed or co-ground with each other. Powder metal compositions according to the present invention are particularly useful in applications where the pre-sintered density of the pressed metal part is less than or equal to about 6.9 g/cm³.

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Description**BACKGROUND OF INVENTION**

5 **[0001]** Field of Invention

[0002] The present invention relates to substantially dry powder metal compositions and methods of making and using the same.

[0003] Description of Related Art

10 **[0004]** Powder metal compositions are frequently used to produce metal parts in applications wherein casting, forging or other metal processing techniques are not cost effective. The fabrication of parts using powder metal compositions includes the steps of placing the powder metal composition in the cavity of a mold, pressing the powder metal composition to form a green compact, removing the green compact from the cavity, and firing the green compact to burn out any organic material and densify and consolidate the metal powder into a final part.

15 **[0005]** Lubricants are employed in pressed powder metallurgy, particularly during the pressing step when the powder is compressed in the cavity to form the green compact. External lubricants, which facilitate the removal of the green compact from the cavity after pressing by ejection, are typically sprayed onto the walls of the cavity prior to filling the cavity with the powder metal composition. Internal lubricants are mixed with the powder metal composition to facilitate slippage of the individual metal particles against each other so that the pressing forces are spread uniformly and the density of the resulting green compact can be made to be as uniform as possible throughout its cross-section.

20 **[0006]** The use of external lubricants is time-consuming. Furthermore, it is often difficult to apply a uniform coating of a liquid external lubricant to the cavity walls, particularly when fabricating complex parts. To reduce and/or eliminate the need for external lubricants, many powder metal compositions are formulated to contain relatively high amounts of one or more internal lubricants. An increase in the amount of internal lubricant(s) present in the composition tends to improve lubricity, which is often measurable in terms of the ejection force necessary to remove the pressed metal part from the die. This approach, while effective at reducing the need for external lubricants, tends to adversely affect the powder metal composition and metal part making process.

25 **[0007]** For example, the presence of relatively high amounts of internal lubricant in a powder metal composition tends to reduce the flow characteristics of the powder metal composition into the mold cavity, thereby reducing the rate at which the pressing operation can proceed. Furthermore, the presence of relatively high amounts of internal lubricants detrimentally affects the density of the green compact (sometimes referred to as "green density" or "green strength"). Thus, in conventional powder metal compositions there is a tradeoff between lubricity and green strength. Furthermore, the presence of relatively high amounts of internal lubricants requires a longer and more complex heating cycle during sintering to remove the larger amount of organic material present. Thus, the use of relatively high amounts of internal lubricants tends to contribute to low final density in the metal part, protracted furnace time, and can lead to the formation of cracks and blisters during firing.

30 **[0008]** Hammond, Pub. No.: US 2007/0048166 A1 (U.S. App. Ser. No. 11 /162,058), which is hereby incorporated by reference in its entirety, discloses substantially dry powder metal compositions that comprise base metal particles, a lubricant that transforms from a solid phase material to a viscous, liquid phase material during pressing, and a micronized deformable solid material. In such compositions, an amount of the micronized deformable solid material that is calculated to fill at least a portion of the void space between the base metal particles during pressing is added to the composition. The presence of the calculated amount of the micronized deformable solid material in such compositions allows at least a portion of the lubricant to migrate as a viscous liquid phase material to the interface between the surface of the green compact and the wall of the mold cavity and thereby provide lubrication that reduces the ejection force necessary to remove the green compact from the mold cavity. Such powder metal compositions are particularly useful in applications where the pre-sintered density of the pressed metal part is relatively high (e.g., greater than about 6.9 g/cm³).

BRIEF SUMMARY OF THE INVENTION

35 **[0009]** Powder metal compositions according to the present invention are particularly useful in applications where the pre-sintered density of the pressed metal part is less than or equal to about 6.9 g/cm³. Powder metal compositions according to the present invention comprise a substantially dry blend of base metal particles and a pressing aid. The pressing aid comprises a micronized deformable solid material and a high melt point lubricating material. The method of forming powder metal compositions according to the invention comprises mixing or co-grinding the micronized deformable solid material and the high melt point lubricating material together to form the pressing aid, and dry-mixing the pressing aid and the base metal particles together to form a substantially dry, substantially homogeneous mixture. The amount of the pressing aid in the powder metal composition need not be calculated based on the void space between the base metal particles after pressing. Instead, fixed, predetermined additions of the pressing aid can be used, typically within the range of from about 0.5% by weight to about 1.5% by weight of the substantially dry powder metal composition.

[0010] Powder metal compositions according to the invention can be used to form pressed metal parts having a pre-sintered density of less than or equal to about 6.9 g/cm^3 that have excellent green strength, and resist cracking, chipping and damage due to handling prior to sintering. The powder metal compositions according to the invention are environmentally friendly, provide excellent lubricity, minimize die wear, exhibit good compressibility and allow for the elimination of zinc-containing lubricants.

[0011] The foregoing and other features of the invention are hereinafter more fully described and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative, however, of but a few of the various ways in which the principles of the present invention may be employed.

DETAILED DESCRIPTION OF THE INVENTION

[0012] As noted above, powder metal compositions according to the present invention comprise a substantially dry blend of base metal particles and a pressing aid. The base metal particles in the powder metal compositions according to the present invention can comprise relatively pure elemental metals, alloys of two or more metals, alloying elements and compounds and/or physical blends or mixtures thereof. Preferred base metal particles for use in the invention include, but are not limited to, iron and steel powders, stainless steel powders, nickel powders, copper powders and brass powders. Such metal powders are commercially available from a variety of sources in a variety of sizes and surface morphologies (e.g., flakes and spheres).

[0013] It is believed that the principles of the invention can also be applied to other pressable inorganic powders (e.g., ceramic particles, intermetallic particles, oxides, carbides etc.). U.S. Pat. No. 6,093,761, from col. 10, line 27 to col. 11, line 20, is hereby incorporated by reference for its teachings relative to the composition of pressable inorganic powders.

[0014] The pressing aid comprises a micronized deformable solid material and a high melt point lubricating material, which have been pre-mixed or co-ground with each other. In the preferred embodiment of the invention, the pressing aid comprises from about 5% to about 40% of the high melt point lubricating material, with the balance being occupied by the micronized deformable solid material. More preferably, the pressing aid comprises about 10% to about 30% of the high melt point lubricating material, with the balance being occupied by the micronized deformable solid material. In the presently most preferred embodiment of the invention, the pressing aid comprises about $15\% \pm 2.5\%$ of the high melt point lubricating material, with the balance being occupied by the micronized deformable solid material.

[0015] The term "high melt point lubricating material" refers to conventional high temperature lubricants such as metal carboxylates and waxes, which are sometimes used alone or in combination with graphite. The preferred high melt point lubricating material for use in the invention is N,N'-ethylenebisstearamide wax (hereinafter "EBS wax").

[0016] The micronized deformable solid material is preferably a Fischer-Tropsch wax that has a high degree of oxidation. The presently most preferred highly oxidized Fischer-Tropsch wax for use in the invention is a highly oxidized polymethylene wax. Polymethylene wax is soft, which necessitates that it be milled under cryogenic conditions in order to obtain particles having a very fine diameter (e.g., $D_{50} < 40 \mu\text{m}$). While it is possible to mix the fine diameter particles of the micronized deformable solid material and the high melt point lubricating material together to form the pressing aid, it is advantageous to co-grind the materials cryogenically to achieve a particles having a very fine diameter ((e.g., $D_{50} < 40 \mu\text{m}$). In such circumstances, the starting materials are preferably in the form of small pellets or beads. Cryogenically co-grinding a mixture of small pellets or beads of EBS wax and polymethylene wax tends to produce a pressing aid in which the polymethylene wax is surface coated with the EBS wax.

[0017] Micronized polymethylene wax is very deformable under conventional powder metal pressing conditions. It does not react with the base metal particles, nor does it react with or adversely affect the lubrication ability of the high melt point lubricating material (e.g., EBS wax). In addition, polymethylene wax can be effectively removed from green compacts using conventional preheating and sintering cycles. It will be appreciated that micronized deformable solid materials other than polymethylene wax may be used in the invention provide such materials do not interfere with the effectiveness of the lubricant or degrade the properties of the final metal part obtained after sintering.

[0018] Liquid lubricants and lubricants containing zinc (e.g., zinc stearate) are preferably not used in the composition. Furthermore, it is not necessary for the pressing aid to include a lubricant composition that transforms from a solid phase material to a viscous liquid phase material when the powder metal composition is pressed to form the green compact. Such a lubricant composition is described in U.S. Pat. No. 6,679,935, which is hereby incorporated by reference in its entirety, and is a component of the powder metal compositions disclosed in Hammond, Pub. No.: US 2007/0048166 A1 (U.S. App. Ser. No. 11/162,058).

[0019] The micronized deformable solid material fills some of the void space between the compressed base metal particles in the green compact, allowing at least a portion of the high melt point lubricating material and the deformable solid to interface between the surface of the green compact and the walls of the mold cavity where it can serve as a lubricant that reduces the ejection force necessary to remove the green compact from the mold cavity. Furthermore, the presence of the micronized deformable solid material in the pressed green compact has the added benefit as functioning

as a binder, which aids in maintaining and enhancing the green strength of the green compact. Thus, the micronized deformable solid material comprises a material that: (1) does not interfere with the powder metal composition compaction process; (2) deforms and slides against the die wall; and (3) provides sufficient lubrication between the surface of the green compact and the walls of the mold cavity to allow the green compact to be ejected from the mold using minimal ejection force.

[0020] The pressing aid is preferably mixed with the dry base metal particles and other optional alloying and/or processing components of the powder metal composition as a solid phase material, and continues to remain as a solid phase material under conventional mold cavity filling conditions. In the pressing step, the high melt point lubricating material remains as a solid phase material that allows the individual base metal particles to slide relatively to each other and efficiently compact together, taking up less volume in the mold cavity and thereby reducing internal void space in the green compact.

[0021] The amount of the pressing aid present in the composition need not be calculated as a function of void space between the base metal particles after pressing, as is disclosed in Hammond, Pub. No.: US 2007/0048166 A1 (U.S. App. Ser. No. 11/162,058). On the contrary, the powder metal compositions according to the invention allow for the use of predetermined, fixed additions of the pressing aid. The amount of the pressing aid present in the composition is preferably the least amount sufficient: (1) to facilitate the efficient compaction of the base metal particles during pressing; (2) to facilitate ejection of the green compact from the mold cavity after pressing; and (3) to achieve a desired green strength in the pressed part. Predetermined loadings of the pressing aid within the range of from about 0.5% by weight to about 1.5% by weight are typically suitable. More preferably, the predetermined loadings are within the range of from about 0.75% by weight to about 1.25% by weight, or about 1.0% by weight.

[0022] Surprisingly, increasing the amount of the pressing aid used in the pressed metal powder composition tends to improve both green strength and lubricity. This is highly unusual inasmuch as improvements in lubricity typically are obtained at the expense of green strength in conventional powder metal compositions. It will be appreciated that a higher loading of the pressing aid can be used for complex metal parts, parts requiring a higher green strength and/or parts having a greater surface area than simple parts having a minimal surface area.

[0023] Powder metal compositions according to the invention can further optionally comprise one or more additives such as, for example, alloying materials (e.g., graphite and/or particles of alloying metals), which are sometimes present in pressed powder metal compositions. The base metal particles, the pressing aid, and any optional additives are blended together to create a substantially homogenous powder metal composition. Mixing assures that the pressing aid and optional additives are evenly distributed throughout the base metal particles so that a green compact having uniform density and structure is obtained subsequent to pressing.

[0024] The present invention provides many advantages and benefits over conventional powder metal compositions and methods. No special set up is required. The powder metal compositions can be used in conventional powder metallurgy dies and equipment. Pressed metal parts exhibiting a higher green strength and a lower ejection force can be obtained. The elimination of zinc-based lubricants can also be achieved.

[0025] The pressing aid allows for efficient base metal particle movement and compaction, which equalizes green density. In addition the presence of the high melt point lubricating material in the pressing aid makes the powder metal composition less subject to packing or caking during storage and/or transportation. The pressing aid minimizes micro-cracking and reduces the risk of molding cracks. The micronized deformable solid material and the high melt point lubricating material are formed of components that decompose at different temperatures, which allows for a staggered or staged burn out. Sintered parts exhibit excellent dimensional stability.

[0026] The following examples are intended only to illustrate the invention and should not be construed as imposing limitations upon the claims.

EXAMPLE 1

[0027] A Fischer-Tropsch wax, namely, SASOLWAX A1, was obtained from Sasol Wax of South Africa. The SASOLWAX A1 material was an odorless, white to off-white water-insoluble powder having a drop melting point of 102°C, a density at 25°C of 0.90 g/cc, and an acid value (ASTM D 1386/7) of 27-29 mg KOH/g, indicating a high level of oxidation. Penetration was carried out at 25°C according to ASTM D1321 to produce granules having a dimension of about 4.0 mm to about 8.0 mm. The granules were then crushed to obtain particles.

[0028] EBS wax, namely ACRAWAX, was obtained from Lonza. The particles of SASOLWAX A1 and EBS wax were mixed at a weigh ratio of 85 parts SASOLWAX A1 to 15 parts EBS wax and then cryogenically co-milled to obtain a pressing aid having a D_{50} of less than about 40 μm .

EXAMPLE 2

[0029] Powder Metal Compositions ("PMC") A and B were formed by blending the constituents shown in weight percent

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in Table 1 together in a V-mixer to form a homogeneous mixture:

Table 1

	PMC A	PMC B
Copper ⁽¹⁾	1.50%	1.50%
Graphite ⁽²⁾	0.30%	0.30%
ACRAWAX ⁽³⁾	0.75%	--
Pressing Aid ⁽⁴⁾	--	1.00%
ponge Iron ⁽⁵⁾	97.45%	97.20%

[0030] Notes: (1) the Copper was obtained from ACuPowder International, LLC of Union, NJ; (2) the Graphite was ASBURY PM9, which was obtained from Asbury Carbons of Asbury, NJ; (3) the ACRAWAX is believed to be a mixture of N,N'-ethylenebisstearamide wax and stearic acid that is commercially available from Lonza Inc. of Allendale, New Jersey; (4) the Pressing Aid was produced in Example 1 ; and (5) the Sponge Iron was Atomet 28, which was obtained from Quebec Metal Powders of Quebec, Canada.

[0031] PMC A and PMC B were each separately pressed until a green part having a 6.6 g/cm³ green density was obtained. Pressing was performed at 140°F to simulate shear energy during conventional pressing. The compaction force in tons per square inch ("TSI Required") to obtain a green part exhibiting a 6.6 g/cm³ green density, the Peak Ejection Force (in ft.-lbs) needed to free the part from the die cavity, the Slide Force (in ft.-lbs) needed to eject the part from the die cavity, the Green Strength of the pressed part (in pounds per square inch) and the Dimensional Change (in %) in the part upon pressing are reported in Table 2 below:

Table 2

	PMC A	PMC B
TSI Required	31	28
Peak Ejection Force	1,931	1,675
Slide Force	1,491	1,100
Green Strength	1,756	2,096
Dimensional Change	0.13	0.11

EXAMPLE 3

[0032] Powder Metal Compositions ("PMC") C, D and E were formed by blending the constituents shown in weight percent in Table 3 together in a V-mixer to form a homogeneous mixture:

Table 3

	PMC C	PMC D	PMC E
Copper ⁽⁶⁾	2.0%	2.0%	2.0%
Graphite ⁽⁷⁾	0.50%	0.50%	0.50%
Zinc Stearate ⁽⁸⁾	0.85%	--	--
Pressing Aid ⁽⁹⁾	--	0.75%	1.0%
Sponge Iron ⁽¹⁰⁾	96.65%	96.75%	96.5%

[0033] Notes: (6) the Copper was obtained from ACuPowder International, LLC of Union, NJ; (7) the Graphite was ASBURY PM9, which was obtained from Asbury Carbons of Asbury, NJ; (8) the Zinc Stearate was conventional powder metallurgy grade zinc stearate; (9) the Pressing Aid was produced in Example 1 ; and (10) the Sponge Iron was Atomet 25, Quebec Metal Powders of Quebec, Canada.

[0034] PMC C, PMC D and PMC E were each separately pressed until a green part having a 6.6 g/cm³ green density

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was obtained. Pressing was performed at 144°F to simulate shear energy during conventional pressing. The compaction force in TSI Required to obtain a green part exhibiting a 6.6 g/cm³ green density, the Peak Ejection Force (in ft.-lbs) needed to free the part from the die cavity, the Slide Force (in ft.-lbs) needed to eject the part from the die cavity, the Green Strength of the pressed part (in PSI) and the Dimensional Change (in %) in the part upon pressing are reported in Table 4 below:

Table 4

	PMC C	PMC D	PMC E
TSI Required	32	30	29
Peak Ejection Force	2,056	1,955	1,572
Slide Force	1,733	1,300	1,125
Green Strength	2,036	2,081	2,657
Dimensional Change	0.16	0.13	0.13

EXAMPLE 4

[0035] Powder Metal Compositions ("PMC") F, G and H were formed by blending the constituents shown in weight percent in Table 5 together in a V-mixer to form a homogeneous mixture:

Table 5

	PMC F	PMC G	PMC H
Graphite ⁽¹¹⁾	0.85%	0.85%	0.85%
Zinc Stearate ⁽¹²⁾	0.75%	--	--
Pressing Aid ⁽¹³⁾	--	0.75%	1.0%
Water Atomized Steel ⁽¹⁴⁾	98.40%	98.40%	98.15%

[0036] Notes: (11) the Graphite was ASBURY PM9, which was obtained from Asbury Carbons of Asbury, NJ; (12) the Zinc Stearate was conventional powder metallurgy grade zinc stearate; (13) the Pressing Aid was produced in Example 1; and (14) the Water Atomized Steel was A1001, Quebec Metal Powders of Quebec, Canada.

[0037] PMC F, PMC G and PMC H were each separately pressed until a green part having a 6.6 g/cm³ green density was obtained. Pressing was performed at 140°F to simulate shear energy during conventional pressing. The compaction force in TSI Required to obtain a green part exhibiting a 6.6 g/cm³ green density, the Peak Ejection Force (in ft.-lbs) needed to free the part from the die cavity, the Slide Force (in ft.-lbs) needed to eject the part from the die cavity, the Green Strength of the pressed part (in PSI) and the Dimensional Change (in %) in the part upon pressing are reported in Table 6 below:

Table 6

	PMC F	PMC G	PMC H
TSI Required	32	30	31
Peak Ejection Force	2,042	2,024	1,738
Slide Force	1,733	1,283	1,058
Green Strength	1,140	2,276	2,520
Dimensional Change	0.14	0.12	0.13

EXAMPLE 5

[0038] Powder Metal Compositions ("PMC") J and K were formed by blending the constituents shown in weight percent in Table 7 together in a V-mixer to form a homogeneous mixture:

Table 7

	PMC J	PMC K
ACRAWAX ⁽¹⁵⁾	1.00%	--
Pressing Aid ⁽¹⁶⁾	--	1.00%
Stainless Steel ⁽¹⁷⁾	99.00%	99.00%

[0039] Notes: (1 5) the ACRAWAX is believed to be a mixture of N,N'-ethylenebisstearamide wax and stearic acid that is commercially available from Lonza Inc. of Allendale, New Jersey; (1 6) the Pressing Aid was produced in Example 1; and (17) the Stainless Steel was 316L, was obtained from Hoeganaes Corporation of Cinnaminson, NJ.

[0040] PMC J and PMC K were each separately pressed until a green part having a 5.5 g/cm³ green density was obtained. Pressing was performed at 140°F to simulate shear energy during conventional pressing. The compaction force in TSI Required to obtain a green part exhibiting a 5.5 g/cm³ green density, the Peak Ejection Force (in ft.-lbs) needed to free the part from the die cavity, the Slide Force (in ft.-lbs) needed to eject the part from the die cavity, the Green Strength of the pressed part (in PSI) and the Dimensional Change (in %) in the part upon pressing are reported in Table 8 below:

Table 8

	PMC J	PMC K
TSI Required	FAIL ⁽¹⁸⁾	21
Peak Ejection Force	FAIL ⁽¹⁸⁾	1,115
Slide Force	FAIL ⁽¹⁸⁾	1,050
Green Strength	FAIL ⁽¹⁸⁾	1,333
Dimensional Change	FAIL ⁽¹⁸⁾	0.12

[0041] Note: (18) no data could be obtained because the composition would not hold together.

[0042] Additional advantages and modifications will readily occur to those skilled in the art. Therefore, the invention in its broader aspects is not limited to the specific details and illustrative examples shown and described herein. Accordingly, various modifications may be made without departing from the spirit or scope of the general inventive concept as defined by the appended claims and their equivalents.

Claims

1. A powder metal composition comprising:

base metal particles; and
a pressing aid;

wherein the pressing aid comprises from a pre-mixed or co-ground blend of a micronized deformable solid material and a high melt point lubricating material, and
wherein the base metal particles, the pressing aid and any optional additives are physically mixed together to form a substantially homogeneous flowable dry powder.

2. The powder metal composition according to claim 1 wherein the pressing aid comprises from about 95% to about 60% of the micronized deformable solid material and from about 5% to about 40% of the high melt point lubricating material.

3. The powder metal composition according to claim 2 wherein the pressing aid is present in an amount of from about 0.5% to about 1.5% by weight of the powder metal composition.

4. The powder metal composition according to claim 1 wherein the micronized deformable solid material is a Fischer-

Tropsch wax.

5. The powder metal composition according to claim 1 wherein the Fischer-Tropsch wax is highly oxidized polymethylene wax.

6. The powder metal composition according to claim 1 wherein the high melt point lubricating material is N,N'-ethylene bis-stearamide wax.

7. The powder metal composition according to claim 1 wherein the micronized deformable solid material and the high melt point lubricating material are co-ground together to a D₅₀ of less than about 40 μm.

8. The powder metal composition according to claim 1 wherein the powder metal composition does not include any zinc-containing lubricants.

9. The powder metal composition according to claim 1 wherein the base metal particles are selected from the group consisting of pure elemental metals, alloys of two or more metals and physical blends or mixtures of two or more thereof.

10. The powder metal composition according to claim 1 wherein the base metal particles are one or more selected from the group consisting of iron powders, steel powders, stainless steel powders, nickel powders, copper powders and brass powders.

11. A method of forming a green compact comprising:

providing a powder metal composition comprising base metal particles and a pressing aid, wherein the pressing aid comprises from a pre-mixed or co-ground blend of a micronized deformable solid material and a high melt point lubricating material, and wherein the base metal particles, the pressing aid and any optional additives are physically mixed together to form a substantially homogeneous flowable dry powder;

placing the powder metal composition into a mold cavity;

pressing the powder metal composition in the mold cavity to form a green compact; and
ejecting the green compact from the mold cavity.

12. A method of forming a powder metal composition comprising:

pre-mixing or co-grinding a micronized deformable solid material and a high melt point lubricating material together to form a pressing aid; and

physically mixing base metal particles with the pressing aid to form a substantially homogeneous flowable dry powder.

13. The method according to claim 12 further comprising:

preselecting an amount of the pressing aid to be present in the powder metal composition within the range of 0.5% to about 1.5% by weight of the powder metal composition.

14. The method according to claim 13 wherein the preselected amount is determined in view of a desired lubricity and green strength of a metal part to be formed by pressing the powder metal composition to a density of less than or equal to about 6.9 g/cm³, wherein both the green strength and lubricity increase as the preselected amount increases.

REFERENCES CITED IN THE DESCRIPTION

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