LUBRICANT FOR POWDER METALLURGY

Inventors: René Lindenau, Radevormwald (DE); Lars Wimbert, Schwelm (DE)

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ABSTRACT
A lubricant for use in a powder mixture is disclosed. This lubricant contains carnauba wax and at least one plant- or animal-based fat. A powder mixture using this lubricant can be used to compact green parts have higher densities and helps to improve the life of the tools compacting the powder.
LUBRICANT FOR POWDER METALLURGY
CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application is a continuation application of PCT application serial number PCT/EP2010/001302 entitled “Lubricant for Powder Metallurgy” filed on Mar. 3, 2010 which claims priority to German patent application number 10 2009 013 021.7 filed on Mar. 16, 2009. The contents of both of these applications are incorporated by reference as if set forth in their entirety herein.

BACKGROUND

[0002] This disclosure relates to a lubricant for powder metallurgy as well as to its use and a sinterable powder mixture that includes the lubricant.

[0003] Lubricants for powder metallurgy are widely used in the manufacture of sintered parts. These sintered parts are often used in the automotive industry and, frequently, as components in engine and transmission systems. Among other things, one difficulty in the manufacture of sintered parts is obtaining a sintered part with a high density. Typically, a preform is first compacted from a sinterable powder in one or more steps to form a green body. This green preform is then sintered in a protective atmosphere during a second sintering step and may be subsequently sized or “coined” to create a strong and dimensionally accurate sintered part.

[0004] The density of sintered parts produced in this way largely depends upon the green density of the preform (also referred to as pressed density) that is achieved during the first compaction step. It is therefore generally desirable to have green bodies with relatively high densities after the compaction. In addition, the final density of a sintered part may be further increased by sizing or coining operations. Furthermore, sizing or coining may be used to improve the geometrical accuracy of the component as needed.

[0005] The high compression pressures commonly used in the state of the art for the manufacture of high-density green bodies result in a high degree of stress on the compaction punches and, moreover, lead to increased friction between the green preform and the die during ejection of the preform from the tool die after compaction. As a result, higher ejection forces must be applied to eject the preform which pose the risk of an undesired local re-densification and the formation of cracks in the green body. It is therefore generally known in the art that a lubricant may be applied to the surface of the punch and/or a suitable lubricant may be added directly to the powder mixture as a binder and to reduce the force for ejection.

[0006] A pressing additive for a sinterable powder mixture is known from DE 102 44 486 A1. This pressing additive contains 20% to 60% by weight a polyglycol and 40% to 75% by weight a montan wax.

[0007] Moreover, lubricants have also been used in sintering operations in order to reduce the forces on the sintering tool, thereby reducing the wear of the sintering tool, and to increase the density of the part, especially in areas near the surface. Generally, mineral oil-based sintering lubricants are used.

[0008] There are significant disadvantages to the lubricants currently in use, especially in the case of those used in metal powder mixtures as pressing additives or sizing lubricants. Many lubricants and/or stearates have skin-irritating or allergenic properties because of the high content of mineral oil or other oil-like substances. In addition, some of the previously used agents can no longer be used as a result of more stringent legal requirements.

SUMMARY

[0009] A lubricant is disclosed that has lubricating qualities similar to known lubricants, but without the handling considerations and constraints of existing lubricants. This lubricant for powder metallurgy comprises a carnauba wax and at least one plant- or animal-based fat. A carnauba wax is a vegetable wax with a density usually in the range of 0.910 to 0.990 g/cc and a melting point in the range of approximately 83°C to approximately 86°C. Carnauba wax is primarily obtained from the leaves of the Brazilian palm tree, Copernicia prunifera (carnauba wax palm). By way of example, carnauba wax contains approximately 85% esters of wax acids by weight, w-hydroxycarboxylic acids and/or cinnamic acids with wax alcohols and diols. Additionally, carnauba wax also contains approximately 3% to approximately 5% free wax acids by weight, especially carnauba and cerotic acids and, in addition, alcohols and diols, hydrocarbons and minerals. Mixtures of different kinds of carnauba waxes may be used. The carnauba waxes may have an iodine value in a range of approximately 8.5 to approximately 10.5. The acid value of the carnauba waxes may be in the range of approximately 1 to approximately 4 and the saponification value may be in the range of approximately 70 to approximately 83.

[0010] The plant- or animal-based fats are triglycerides. The term fat as used herein is synonymous with the term oil, so that it is possible to speak comprehensively of the group comprising fats and oils. These fats and oils largely comprise mixed glycerol esters of higher fatty acids with an even number of carbon atoms, whereby animal fats may also contain fatty acids with an odd number of carbon atoms. The fat may be selected from a group that includes one or more vegetable fats, prepared separately or as a mixture. The fat contained in the lubricant according to the invention may contain at least 6% oleic acid by weight in some forms and at least approximately 10% oleic acid by weight in other forms. Preferably, the quantity of oleic acid is in the range of approximately 6% to approximately 65% by weight, based on the total amount of fat respectively. The fat contained in the lubricant according to the invention has an iodine value of at least 40, and more preferably of at least 80. In an further embodiment, the fat in the lubricant has a saponification value of at least approximately 150 mg KNO3/g, and more preferably of at least approximately 200 mg KNO3/g.

[0011] This lubricant for powder metallurgy is primarily produced for the manufacture of sintered parts. Sintered parts are to be understood as parts that are manufactured entirely of a sinterable material or partly of a sinterable material (as is the case for composite parts). In some forms, a first portion of such a composite part can be manufactured, for example, of a mixture containing aluminum or iron, and a second portion that is connected to the first portion may be made of another material, e.g., cast iron, sintered or solid or manufactured of solid cast aluminum. In other forms, the composite part may have a sintered layer on surface(s) of a base material. In some forms, the sintered parts can be sized or coined using the lubricant and/or heat treated.

[0012] The sintered parts are primarily manufactured from a mixture comprising at least one metallic material and/or plastic material and at least one lubricant for powder metallurgy. Sinterable materials are, as used herein, powders or
powder mixtures made of metallic, ceramic and/or plastic components; for example, low alloy steels, chromium-nickel steels, bronze, nickel-based alloys such as Hastalloy, Inconel, metal oxides, metal nitrides, metal silicides or the like, and further, powders or mixtures containing aluminum, whereby the mixtures may also contain high-melting components such as platinum or the like. The powders and their particle sizes depend upon the particular application. By way of example, powders that contain iron are alloys such as 316L, 304L, Inconel 600, Inconel 625, Monel and Hastalloy B, X and C as well as 17-4PH. By way of example, low-alloy steel powders may include, for example, carbonized steel, Distaloy AB, AE, DE and HP (Höganäs AB, Sweden) and Ancorsteel 4300 (Hoe-ganaes Corp., USA). Titanium and/or titanium alloys are also suitable materials, even when mixed with other materials, such as powders containing iron. Furthermore, the metallic material and/or plastic material may include synthetic fibers or fibers such as fibers with a diameter between 0.1 μm to approximately 2 μm and of a length of a few microns up to approximately 50 millimeters. In addition, carbon may be added in the appropriate quantity to some metallic materials (e.g., iron) in order to arrive at the desired alloys. Other additives such as binding agents or the like may also be added. In addition, the sinterable mixture may also contain at least one stabilizing agent and/or at least one anti-agglomeration agent. Furthermore, the sinterable mixture may also contain self-lubricating materials such as MoS₂, WS₂, BN and/or other carbon modifications such as coke, polarized graphite or the like in addition or as an alternative to graphite. In addition, the sinterable mixture may contain aerosols, as well as other additives known to the person skilled in the art, depending upon the particular application.

Some lubricants of the type described have the significant advantage that, with the lubricant, sinterable materials or material mixtures have a compressibility similar to that found when conventional pressing additives are used. Furthermore, some lubricants of the type described can be used as a sizing lubricant by which it is possible to achieve an especially high force for a unilateral calibration on the lower punch, for example, using load cells. Furthermore, the ejection pressures are as low as for lubricants known from the prior art that are, however, more disadvantageous from a handling/environmental perspective.

In one preferred embodiment, at least one of the fats comprised in the lubricant is solid or fluid.

Here, the aforementioned aggregate states are based on a temperature of 20°C, but, depending on the composition and viscosity, a fat that melts at 5°C, for example, may also be referred to as a solid fat. In some embodiments, at least one fat may be a triglyceride, which is a glycerol ester with predominantly saturated fatty acids, meaning that the glycerol ester is comprised of at least two fatty acids.

In one preferred embodiment, the lubricant includes at least one fat that is a solid at a temperature of 20°C. In addition to carnauba wax. In another preferred embodiment, the lubricant according to the invention includes at least one fat that is a solid at a temperature of 20°C. In at least one fat that is liquid at 20°C, in addition to carnauba wax.

At least one fat in the lubricant for powder metallurgy may be selected from a group that includes rapeseed oil, coconut oil, soya oil, linseed oil, palm oil and/or fat, sunflower oil, walnut oil, hazelnut oil, olive oil, castor oil, tallow and/or fish oil and derivatives of the aforementioned substances. Particularly suitable derivatives include hydrogenated or oxygenated compounds of the aforementioned substances. Mixtures of the specified substances may be used.

The lubricant may contain at least 50% carnauba wax by weight and at least 10% by weight of one of the vegetable fats that is a solid at a temperature of 20°C, where the weight percentages are calculated based on the total amount of the lubricant.

The lubricant may contain carnauba wax in an amount of 65% to 90% by weight and the at least one solid vegetable fat in an amount of 10% to 35% by weight, each calculated on the basis of the total weight of the lubricant. In some forms, the lubricant may contain no additional substances besides carnauba wax and the minimum of one solid vegetable fat.

The lubricant may contain at least approximately 5% carnauba wax by weight, at least approximately 20% by weight of a first vegetable fat that tends to be solid at a temperature of 20°C, and at least approximately 40% by weight of a second vegetable fat that is liquid at a temperature of 20°C, whereby the weight percentages are calculated based on the total quantity of the lubricant. In this embodiment, the lubricant may contain carnauba wax in an amount of approximately 6% to approximately 15% by weight, the first vegetable fat in an amount of approximately 30% to approximately 45% by weight and the second vegetable fat in an amount of approximately 45% to approximately 65% by weight. The lubricant may, in some forms, contain no further substances in addition to a second liquid vegetable fat, a first vegetable fat and carnauba wax. That embodiment of the lubricant is especially suitable for use as a sizing lubricant, as no drying step is required before sizing, as is often the case for sizing lubricants that contain solvents as is found in prior art. The lubricant as further described above, which contains a higher proportion of carnauba wax, is preferably used as a pressing additive and is more preferably mixed directly into a powder mixture.

Insofar as ranges or number ranges are indicated, it should be noted that the particular upper and lower values of these ranges are not absolute values. Rather, some deviations from the numerically defined as upper and lower limits may be made where the application so demands. Here, variances within a deviation range of up to 5% from the indicated numeric value of the upper and/or lower level are possible.

The present invention also pertains to the use of a lubricant as defined above, as an additive to a sinterable powder mixture, or in other words, as a pressing additive or in some alternate forms, as a sizing lubricant in powder metallurgy.

Finally, the present invention also pertains to a mixture for the manufacture of sintered parts comprising at least one metallic material and/or plastic material and at least one lubricant for powder metallurgy as defined above. A sinterable powder mixture will be used that further contains at least one plastic and/or metallic powder material in addition to at least one disclosed lubricant. The mixture for the manufacture of sintered moldings according to the invention primarily contains 0.1% to 2% of the lubricant by weight, calculated on the basis of the total weight of the mixture. In addition, aside from the aforementioned metallic and/or plastic materials, including by way of mixtures, the sinterable powder mixture may also contain further additives, such as aerosols, graphite, self-lubricating materials, binding agents, and so forth.

These and still other advantages of the invention will be apparent from the detailed description and drawings. What
follows is merely a description of some preferred embodiments of the present invention. To assess the full scope of the invention the claims should be looked to as these preferred embodiments are not intended to be the only embodiments within the scope of the claims.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a chart depicting the green density of compacted parts using convention Licowax C lubricant and one formulation of the disclosed lubricant at various compaction pressures.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Pressing Additive

A lubricant that is used as a pressing additive is provided by a mixture of 16.7% coconut oil by weight and 83.3% carnauba wax by weight. The coconut oil was still a solid fat at a temperature of 20°C with a saponification value of 255 mg KOH/g to 260 mg KOH/g. The coconut oil was purchased under the trade name Palmim, which is manufactured by the company Peter Kölln KGaA, Elmsborn, Germany. The carnauba wax that was used was obtained under the type designation 7170, manufactured by the company Willy Benecke GmbH, Hamburg, with a melting range of 78°C to 90°C, and an acid value of 2 to 10 as well as an ester value of 70 to 82. The carnauba wax was available in powder form.

The coconut oil, which was available in block form, and the carnauba wax, which was available in powder form, can be melted together in the indicated amounts for the manufacture of the lubricant. However, in other forms of production, they can also be mechanically mixed together as solids (after reconstituting the coconut oil into a powder or pellet form). If the lubricant is manufactured by melting together the components, the melt may be allowed to cool and then may be ground or atomized. The lubricant that was produced in this way and that is usable as a pressing additive was obtained as a solid powder at a temperature of 20°C.

The prepared lubricant was added to a sintenable metallic powder as a pressing additive. The base powder was a water-atomized iron powder available under the trade name 1000 BMn, manufactured by Hoeganaes Corporation, Cincinnati, USA. To this base powder was added 2% copper by weight in powder form and 0.6% graphite by weight available under the trade name UF-4, manufactured by Graphit Kropfmüller AG, Hauenberg, Germany. The pressing additive was added to this powder mixture in an amount of 0.6% by weight of the lubricant and the mixture was homogenously mixed. The sinterable powder mixture thus prepared was placed in a conventional compaction press and uni-axially pressed into bushings with an outside diameter of 14.3 mm, an inside diameter of 9 mm, and a height of 13.3 mm at different pressures and a mold temperature of 25°C as well as an M/Q ratio of 10 (lateral surface to cross section ratio).

For comparison, a sintenable powder mixture of the aforementioned metal composition was prepared, whereby instead of 0.6% by weight of the stated lubricant being added as a pressing additive, this later preparation used an amount of 0.6% by weight of the pressing additive known from the prior art, Licowax C manufactured by Clariant GmbH, Frankfurt am Main, Germany, which is a bis-stearoylethylene diamine (amide wax).

The density of the produced green bodies was measured at different pressing pressures, both for the powder mixture and for the comparison mixture in accordance with DIN 1503369 (impermeable sintered metals and carbides/ investigation of the density). The density was obtained at pressing pressures of 400 MPa, 500 MPa and 600 MPa.

FIG. 1 shows that when the newly disclosed lubricant was used as a pressing agent, the densities of the produced bushing-shaped green bodies were higher than those achieved when the lubricant known and tested from the prior art, Licowax C, was used. Unlike Licowax C, however, the newly disclosed lubricant is environmentally friendly and cost-effective.

Sizing Lubricant

Another lubricant, this time for sizing, was prepared using 54% rapeseed oil by weight (second vegetable and liquid fat), 36% by weight of a first vegetable fat and 10% carnauba wax by weight. The carnauba wax used here corresponded to that which is used in the lubricant as a pressing additive, described above in greater detail. The rapeseed oil had a melting point of 5°C and, therefore, can be considered a liquid fat in the sense of the present application. The saponification value was 375 mg KOH/g, the iodine value 107, the viscosity, measured at 35°C and measured dynamically, was 39 mPa/s (measured in accordance with DIN 53015). The used first vegetable fat had a melting point of 33°C and a viscosity, measured at 35°C and measured dynamically, of 78 mPa/s (measured in accordance with DIN 53015) and, therefore, can be considered to be a solid fat. The first fat had a saponification value of 380 mg KOH/g and an iodine value of 92 mg KOH/g. The proportion of oleic acid in the first fat was approximately 52% by weight, whereas the oleic acid in the rapeseed oil that was used was present in an amount of approximately 59% by weight. The individual components of the lubricant (which can be used as a sizing lubricant) were mixed at an increased temperature of approximately 80°C in liquid form.

Using the prepared sizing lubricant, bushings were produced out of a water-atomized iron having an inner diameter of 9 mm, an outside diameter of 14.3 mm, and a height of 25 mm. The water-atomized iron was manufactured under the trade name of ASC100.29 manufactured by Hoeganaes AB, Sweden. The aforementioned iron powder was pressed, together with 0.6% Licowax C by weight, into the aforementioned components. The lateral surface to cross section ratio was 19. The green parts obtained were sintered at 1120°C for approximately 20 minutes in a continuous belt oven. Once the manufacturing step of sintering and cooling to room temperature is complete, the bushings obtained in this manner were dipped in the calibration lubricant at 20°C.

After the dipping process and, if necessary, drying step (a processing step which, unlike lubricants known from prior art, is not necessary using the above-described sizing lubricant), the bushings coated with the lubricant were placed in an appropriate sizing tool and a unilaterial sizing was performed using an upper punch with a force of 800 MPa. In so doing, the forces that resulted from the upper punch during sizing were measured using load cells manufactured by Höttinger Baldwin, Darmstadt, Germany. The forces on the lower punch were measured first. After sizing, the samples were then ejected from the lower punch of the sizing tool and the force needed to do so was measured. While the forces measured on the lower punch should be as high as possible during
sizing, the forces measured when the component is ejected from the tool should be as low as possible.

[0035] The lubricant mixture prepared as described above, which can be used as a sizing lubricant, was compared with the conventional lubricant, Multical EJ10 manufactured by Zeller+Gmelin GmbH & Co. KG, Eisingen/Fils, Germany (which in the meantime has been removed from the market), and which is a solvent-containing wax. It was also compared with Rustilo DWX 30 manufactured by Castrol Industrie GmbH, Mönchengladbach, Germany, which is used as a calibration lubricant, and which is a solvent-containing fat.

[0036] The following table, Table 1, compares the measured forces and the manufacturing conditions.

### TABLE 1

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Basis</th>
<th>Product</th>
<th>Drying time [min]</th>
<th>Drying at Temp. [°C]</th>
<th>Tool Temp. [°C]</th>
<th>( P_{\text{Lower punch}} ) [MPa]</th>
<th>( P_{\text{Pus-off}} ) [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zeller + Gmelin</td>
<td>Wax, containing</td>
<td>Wax, containing</td>
<td>720</td>
<td>25</td>
<td>25</td>
<td>579</td>
<td>165</td>
</tr>
<tr>
<td>GmbH &amp; Co. KG</td>
<td></td>
<td>containing</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Castrol Industrie</td>
<td>Fat, containing</td>
<td>Fat, containing</td>
<td>300</td>
<td>25</td>
<td>25</td>
<td>215</td>
<td>515</td>
</tr>
<tr>
<td>GmbH</td>
<td></td>
<td>containing</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Disclosed lubricant</td>
<td>Disclosed</td>
<td>Disclosed</td>
<td>0</td>
<td>25</td>
<td>25</td>
<td>490</td>
<td>185</td>
</tr>
</tbody>
</table>

[0037] As can clearly be seen in the table above, the newly disclosed lubricant, used as a sizing lubricant, shows improved qualities both in terms of the forces measured at the lower punch as well as relative to the measured ejection pressure values, which fall between those of sizing lubricants known from the prior art, and specifically in ranges that are relevant to practical application. Moreover, the newly disclosed sizing lubricant demonstrates extraordinarily environmental friendliness and occupational safety qualities in comparison to the conventional sizing lubricants.

[0038] It should be appreciated that various other modifications and variations to the preferred embodiments can be made within the spirit and scope of the invention. Therefore, the invention should not be limited to the described embodiments. To ascertain the full scope of the invention, the following claims should be referenced.

What is claimed is:

1. A lubricant for powder metallurgy comprising: carnauba wax; and at least one plant- or animal-based fat.
2. A lubricant in accordance with claim 1 wherein the fat is a vegetable fat.
3. A lubricant in accordance with claim 1 wherein the at least one fat contains at least 6% oleic acid by weight.
4. A lubricant in accordance with claim 1 wherein the fat has an iodine value of at least 40.
5. A lubricant in accordance with claim 1 wherein the fat has a saponification value of at least 150 mg KOH/g.
6. A lubricant in accordance with claim 1 wherein the at least one fat is solid or liquid.
7. A lubricant in accordance with claim 1 wherein the fat contains at least one fat that is solid at a temperature of 20°C.
8. A lubricant in accordance with claim 1 wherein the fat contains at least one fat that is solid at a temperature of 20°C and at least one fat that is liquid at a temperature of 20°C.
9. A lubricant in accordance with claim 1 wherein at least one fat is selected from a group consisting of rapeseed oil, coconut oil, soya oil, linseed oil, palm oil or palm fat, sunflower oil, walnut oil, hazelnut oil, olive oil, castor oil, tallow, fish oil, and derivatives of the aforementioned substances.
10. A lubricant in accordance with claim 1 wherein the lubricant comprising at least 50% carnauba wax by weight and at least 10% of a vegetable fat by weight that is solid at a temperature of 20°C, wherein the weight percent are calculated based on the total quantity of the lubricant.
11. A lubricant in accordance with claim 1 wherein the lubricant comprises at least 5% carnauba wax by weight, at least 20% by weight of a first vegetable fat and at least 40% by weight of a second vegetable fat that is liquid at a temperature of 20°C, wherein the weight percent are calculated based on the total quantity of the lubricant.
12. A method of using the lubricant of claim 1 as an additive to a sinterable powder mixture or as a sizing lubricant in powder metallurgy.
13. A mixture for the manufacture of sintered parts comprising at least one lubricant in accordance with claim 1 and at least one metallic material.
14. A mixture in accordance with claim 13 wherein the mixture contains 0.1% to 2.0% lubricant by weight, calculated on the basis of the total weight of the mixture.

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