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POWDER METALLURGY

Leon J. Printz, Detroit, Mich., assignor to Chrysler Corporation, Highland Park, Mich., a corporation of Delaware

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ABSTRACT OF THE DISCLOSURE

A process for producing sintered porous metal objects through the compacting of powder metals at low pressure. A fatty acid and a wax are blended with the powdered metal prior to compacting so as to form a green briquette of sufficient firmness and cohesiveness to withstand normal handling without fracture or crumpling. Prior to sintering, the green briquette is placed under reduced pressure and heated to remove the fatty acid and wax components, thereby avoiding dissociation of these materials at sintering temperatures into substances which react with the powder metal to adversely affect the chemical and physical properties of the desired product. The green briquette is then sintered after removal of the fatty acid and wax.

BACKGROUND OF THE INVENTION

The present invention relates to porous metal objects and to a process for their manufacture involving the compacting and sintering of powder metal. Porous sintered metal articles such as bearings have long been known and have found wide acceptance due to their lubricating effectiveness when impregnated with a lubricant such as oil. Moreover, porous aluminum bearings are finding increasing use due to their excellent heat conductivity, low weight and strength. Accordingly, the balance of the discussion of this invention will be with respect to the manufacture of sintered aluminum and aluminum alloy metal, although it will be appreciated that the invention is applicable to other metal systems such as bronze and copper.

While it is recognized that porous sintered aluminum is an excellent material from which to fabricate bearings, it is also known, that the bearing performance is largely dependent upon the quantity of oil or other lubricant which can be incorporated into the bearing and the ease with which the lubricant can move to the load bearing surfaces. Understandably, the oil absorption and flow is primarily a function of the density of the metal bearing, and it is known that high density bearings, that is, those formed at higher compacting pressures, generally are shorter lived than those formed at lower pressure since they are less porous, contain less oil and offer fewer oil flow paths. Accordingly, certain manufacturers engaged in the powder metal bearing art attempt to provide a highly porous sintered metal bearing by using a very low compacting pressure to briquette the powder metal. Unfortunately, this approach has many problems stemming from the fact that metals, and aluminum in particular, are coated with a protective oxide film which inhibits formation of satisfactory metal to metal bonds. Accordingly, when compacted under low pressure, the resulting green briquette does not have sufficient strength and cohesiveness to permit normal handling during the subsequent sintering and sizing manufacturing operations. Therefore, a high amount of scrappage is encountered unless extremely delicate handling measures are used. Obviously, neither situation is compatible with high volume, low cost production.

To avoid the above difficulties associated with the use of low briquetting or compacting pressures, many in the art have turned to the use of high compacting pressures, i.e., 30 to 75 tons per square inch. The use of such high pressures, however, causes many other problems which largely offset the advantages secured by the increased cohesive strength of the green briquette produced thereby. For example, the use of high pressure has been found to cause the metal to stick to the die wall thereby requiring the need of costly equipment to eject the briquette from the die. Moreover, the high forces generated during ejection have been known to shatter or break away portions of the briquette thus destroying the accuracy of the configuration. Accordingly, the art is repleat with various methods for lubricating the walls of the die to prevent such sticking. Such methods are generally costly and necessitate frequent die cleaning. A more serious problem lies in the fact that high briquetting pressures generally result in high density bearings with low oil absorption capacity. In order to provide for increased oil absorption, a number of methods have been developed for increasing porosity through reduction of the metal oxide component of the bearing. For example, U.S. 2,133,761 teaches to add copper oxide to the green briquette and then reduce the oxide during sintering so as to provide voids in the bearing. However, such oxide reduction techniques are costly and dangerous since they generally required using hydrogen at high temperatures which is an extremely hazardous procedure. Accordingly, it is seen that the use of high briquetting pressures does not provide a commercially attractive process due to the cost of large die and die ejecting equipment and the need for hazardous reduction procedures to create the necessary bearing porosity.

SUMMARY

An object of the present invention, therefore, is to provide an improved porous metal object by the metallurgical techniques involving compacting and sintering of powder metal.

A further object is to provide an improved method of making low density sintered metal objects which overcomes prior difficulties and which is simple and effective.

A particular object of this invention is to provide a method of preliminary compacting, without the use of high pressures, aluminum metal powder briquettes which are adapted to being worked by conventional processes.

Other objects and advantages of the present invention will become apparent from a further reading of the description and the appended claims.

This invention is based on my discovery of certain agents which enable the production of strong, cohesive green powder metal briquettes through the use of low compacting pressures and, if high compacting pressure is employed, eliminate the need for metal oxide reduction to provide suitable porosity and lubricant absorption properties.

The foregoing results are achieved by first blending the powder metal, prior to compacting, with a fatty acid and a wax so as to form a mixture of powder metal, fatty acid and wax and then compacting the mixture into a green briquette of the desired configuration. The green briquette is then vacuum heated to remove the fatty acid and wax components prior to sintering of the briquette. It is essential that substantially all of the fatty acid and wax be removed so as to prevent their dissociation and reaction at sintering temperatures with the metal powder, since such reaction has been found to seriously adversely effect

the chemical and physical properties of the end product. Finally, after such removal, the green briquette is sintered and treated to produce the desired result.

DESCRIPTION

The aluminous metal powders which can be employed in this invention may be of the flake or atomized type and the selection of the form of powder and particle size is dependent upon the use and performance requirements of the end product. While a wide variation in particle size is permissible, the particles should not be larger than will pass through a 35 mesh screen (Tyler Sieve Series). In general, it is desirable to utilize powders of a fine mesh size as in the range of 200, —325 mesh and mixtures of different sizes, as is well known in the art, are frequently advantageous in securing certain properties. The metal powder can consist of low purity aluminum, for example 99 percent, up to the highest purity obtainable, or particles of aluminum base alloys such as are formed by dissolving the alloying metal in molten aluminum, or a mixture of aluminum and the desired alloying elements such as zinc, copper, manganese, tin, lead and magnesium and silicon which are commonly employed in the aluminum-alloy art. Excellent aluminum based bearings have been produced in accordance with this invention from an elemental alloy mixture consisting of, based on weight, 2–5% copper, 1–5% tin, 0–4% lead, 0–1.5% magnesium and the remainder aluminum. In general, aluminum bearings should contain from about 75 to 95 weight percent of aluminum, the remainder essentially being an alloy material. However, where extremely small particle sizes are employed, for example —400 mesh, the aluminum content of the bearing can be as low as 50 weight percent.

Prior to preparing the initial green briquette, the metal powder is blended with a fatty acid and a wax. The fatty acid component should contain at least about 12 carbon atoms and can be either saturated or unsaturated. Preferably, the fatty acid contains from about 12 to 22 carbon atoms and mixtures of such acids can be used. Examples of suitable acids include, lauric acid, palmitic acid, margaric acid, tridecanoic, stearic acid, oleic acid, brassidic acid, arachidic acid, linoleic acid, behenic acid, erucic acid, linolenic acid, elaidic acid, eleostearic acid, lichemic acid, ricinoleic acid, palmitoleic acid, and petroselenic acid. The commercially available flake or powdered form of these acids can be used in this invention. It has been found that the quantity of acid needed in the process of this invention is dictated by the configuration and metal composition of the green briquette and that the suitable amount for any given application can be determined by routine experimentation by one of skill in the art. In general, good results have been obtained when the mixture to be compacted contains from about 0.2 to 2 weight percent of the fatty acid.

The primary criteria for selection of a suitable wax for use in this invention are that it act as a good binder for the metal powder so as to provide substantial cohesive strength in the green briquette and that it be volatilizable at a temperature not exceeding about 345° C. Naturally occurring animal and vegetable waxes as well as synthetic waxes which have a melting point up to about 150° C. have been found to meet these requirements. Representative of waxes which can be employed in this invention are the ester reaction products of high molecular weight fatty acids, such acids having from about 12 to 34 carbon atoms, with compounds containing at least one active hydrogen atom. The term "active hydrogen atom" refers to hydrogen which, because of its position in the molecule, displays activity according to the Zerewitinoff test as described by Kohler in J. Am. Chem. Soc. 49 3181 (1927). The active hydrogen atoms are generally attached to oxygen, nitrogen or sulfur such as —OH, —SH, —NH—, $—NH_2$, $—CONH_2$, —CONHR where R represents an organic radical, $—SO_2OH$, $—SO_2NH_2$, or $—CSNH_2$, and may be part of aliphatic, aromatic, cycloaliphatic or mixed type compounds. Typical of many active hydrogen containing organic compounds which are useful are alcohols such as cetyl alcohol, ceryl alcohol, n-octadecyl alcohol, montanyl alcohol and myricyl alcohol, and polyhydric alcohols such as ethylene glycol, diethylene glycol and polyethylene glycol. Other representative compounds include nonoethanolamine, sulfonilamide, propylenediamine and ethylenediamine. Good results are obtained when the wax is an ester reaction product of a fatty acid having from about 12 to 34 carbon atoms and an alcohol having one or two hydroxyl groups. Examples of such materials are: carnauba wax having a melting point of about 80–87° C. and which is a mixture of the esters of the normal alcohols and fat acids having even numbers of carbon atoms from 24 to 34; beeswax which has a melting point of 60–82° C. and whose composition resembles a carnauba wax except it is mainly composed of 25 and 28 carbon atom acids and alcohols; and spermaceti which is mainly cetyl palmitate and which melts at 42–47° C. Equally good results are also obtained from synthetic waxes such as the amides, nitriles and amines of the higher fatty acids, all of which are familiar materials to those in the art. The amount of wax which is required is variable but it has been found that an amount in the range of about 0.1 to 1 weight percent is generally satisfactory.

The method by which the powdered metal is mixed with the fatty acid and wax components is not material provided that a substantially uniform mixture is obtained. Thus any form of mixing can be employed, such as hand mixing or any of the mechanical methods for uniformly mixing powdered materials. After blending, the mixture of powder metal, fatty acid and wax is compacted into a green briquette. Briquettes having a high degree of firmness and cohesiveness have been formed without the need of external heating through the use of relatively low compacting pressures in the range 2 to 10 tons per square inch. Higher pressures can be employed if desired. Since this invention permits the use of relatively low compacting pressures, such pressures can be exerted by conventional and rather simple means and thus obviate the need for special dies or presses and high pressure equipment. This represents a considerable economy in the production of powder compacts.

Prior to heating the green briquette to sintering temperature, it is necessary that substantially all of the fatty acid and wax components of the briquette be removed since it was found that these components disassociated at temperatures above about 345° C. into constituents which adversely affect the chemical and physical properties of the sintered product. Accordingly, after briquetting, the green briquettes are supported on trays and placed in a vacuum chamber which is then evacuated, for example to a partial pressure of about 50 to 200 microns. After the chamber has been evacuated to the desired vacuum, or with the start of the evacuation step, heat is then applied to the chamber to raise the temperature of the green briquettes to an elevated temperature above the melting point of the wax but not exceeding about 345° C. During the heating, a gas, preferably an inert gas, is bled into the chamber and into direct contact with the briquettes so as to sweep the volatized fatty acid and wax components from the chamber. Bleeding of the gas into the chamber will cause the pressure in the chamber to rise, and it has been found that to insure essentially complete removal of the fatty acid and wax from the briquettes, that is, at least about 95 percent removal, the chamber should be kept at a partial pressure not exceeding about 3500 microns. In the practice of this invention excellent results have been obtained when chamber pressure was in the range of about 200 to 3500 microns and preferably from about 500 to 1500 microns.

The sweep gas should be bled into the chamber at a

rate sufficient to remove the volatized fatty acid and wax components from the vicinity of the briquettes and to prevent back diffusion of these components. Experiments as to the proper rate have shown that excellent results are obtained, for example, at a sweep gas flow rate in the range of about 1000 to 1500 cubic centimeters (c.c.) per minute per briquette at a chamber pressure of 1000 microns. Thus, for example, if the chamber operating at a partial pressure of 1000 microns contained 10 briquettes, a flow rate in the range of 10,000 to 15,000 cc. per minute would be used. In addition to removing the volatiles, the sweep gas provides the desired results of a more uniform heating of the briquette and it has been found that any non-oxidizing gas can be used. As mentioned earlier, it is not necessary in the practice of this invention to provide for reduction of a metal oxide as in many heretofore known processes and, hence, there is no need to use hydrogen or ammonia which is an extremely hazardous procedure at elevated temperatures. The preferred sweep gases for use in this invention are the inert gases such as nitrogen, argon, helium and the like.

The rate at which the briquettes are initially heated to volatize the fatty acid and wax is not critical and it will be understood that the briquettes can be placed in a chamber which is cool and the chamber then brought up to elevated temperatures, or they can be placed directly into a chamber which is already at an elevated temperature. The briquettes should be heated for a sufficient time to permit essentially complete volatization of the fatty acid and wax and this can be easily determined by analysis of the sweep gas coming from the chamber. For example, in producing aluminum bearings by this invention, it was found that the fatty acid and wax were completely volatized in the 20 minute period required to heat the briquettes to a temperature of approximately 320° C.

After the fatty acid and wax components have been removed, the briquettes are heated to a suitable sintering temperature, generally above 500° C., which is determined primarily by the composition of the green briquette. Preferably, the sintering is accomplished by merely increasing the temperature in the chamber while continuing to employ a sweep gas and partial vacuum. However, the sintering operation does not require the use of a sweep gas and partial vacuum and these conditions need not be employed. Likewise, the green briquettes upon removal of the fatty acid and wax components can be transferred to another furnace for sintering. To complete the fabrication of bearings, the sintered metal articles are impregnated with oil as by immersing them in oil in a vacuum chamber. The chamber is then evacuated until air no longer flows out of the bearings and the chamber is returned to atmospheric pressure so as to allow oil to fill the bearing. The bearing is then coined.

The following examples illustrate the process of this invention and the product produced thereby, the invention however not being limited to the specific details thereof.

EXAMPLE I

The process of this invention was used to produce bearings having the following composition by weight: 3–4% copper, 2–5% tin, 0–4% lead, 0.5–0.7% magnesium and 86.3 to 94.5% aluminum. The metal powders used to prepare these compositions had the approximate following particle size distribution:

	Screen Analysis (Tyler Sieve Series)			
	+65 Mesh	+200 Mesh	+325 Mesh	−325 Mesh
Percent:				
Copper	0	9	27	64
Tin	0	1	3	96
Lead	0	1	3	96
Magnesium	0	0	2	98
Aluminum	7	31	17	45

The aluminum had an apparent density of 1.1–1.3 gms./cc., a chemical purity of 99.5% minimum and average particle diameter of 22–26 microns; the magnesium had an apparent density of 0.5–0.6 gms./cc. and a chemical purity of 99.8%; the copper had an apparent density of 0.9–1.1 gms./cc. and a chemical purity of 99.4% minimum; the tin had an apparent density of 2.50–3.60 gms./cc. and a chemical purity of 99.5% minimum; and the lead had an apparent density of 4.75–5.75 gms./cc. and a chemical purity of 99.5% minimum.

EXAMPLE II

This example illustrates the preparation, using the materials defined in Example I, of porous aluminum bearings of the following composition:

Material:	Parts by weight
Aluminum	90.58
Magnesium	0.75
Copper	4.00
Tin	2.67
Lead	2.00
Ethylene distearamide	0.25
Stearic acid	0.75

A 200 pound mixture of the above composition was prepared by adding 1.5 pounds of stearic acid powder to 7.5 pounds of aluminum powder to form a first mixture. This mixture was then stirred to form a uniform blend and passed through a 40 mesh screen into a storage container. The stearic acid which was used had a sieve analysis of 99.5% through 30 mesh, 95% through 100 mesh; a titer of 147–148° F.; an acid value of 198–203; an iodine value of 64–65; and was a stearic acid palmitic acid mixture containing about 83% stearic acid.

Using the same procedure, a second mixture was prepared by adding 0.5 pound of ethylene distearamide to 5 pounds of aluminum powder. These first and second mixtures were then added to 181.15 pounds of aluminum powder along with 8.0 pounds of copper, 4.0 pounds of lead, 5.35 pounds of tin and 1.5 pounds of magnesium. This mixture was then blended for 30 minutes so as to provide for uniform distribution and had a density of approximately 1.3 gm./cc. After blending, the mixture was placed into a die and compacted at a pressure of approximately 4 tons per square inch into green briquettes. No external heating was used during compaction and the briquettes had a density of about 2.25 gm./cc.

The briquettes were then placed loosely into a basket which was then placed on removal shelves of a vacuum retort. Each basket contained approximately 40 briquettes and 14 baskets were arranged in the retort. Vacuum pumps were then started to produce a partial vacuum or pressure in the retort of about 85 microns. Nitrogen was then bled into the chamber and the pressure in the retort rose to about 600 microns. Simultaneously with the start of the nitrogen flow a furnace was lowered about the retort and the briquettes are brought to a temperature of about 315° C. in about 20 minutes. At the end of this period, analysis of the nitrogen sweep gas being exhausted from the retort showed that the stearic acid and ethylene distearamide had been removed from the briquettes and the temperature of the briquettes was then raised to 545° C. and held for approximately 30 minutes while continuing the flow of nitrogen and maintaining the vacuum. At the end of this sintering period, the furnace was raised and the retort was opened after reaching a temperature of about 70° C.

The thus produced sintered bearings, having a length of 0.75 inch, an outside diameter of 1.0 inch and an inside diameter of 0.75 inch, were then impregnated with a Terrestic grade oil, which is a highly refined turbine oil having a high viscosity index and oxidation stability, under a

vacuum of 26 inches of mercury. The bearings were then tested and the following data obtained:

Density ____gm./cc__	2.20/2.35
Oil content ____percent minimum__	15
Compressive strength ____lbs./sq. in__	12,000
Apparent hardness RH	40/50
PV performance*	55,000

*P equals bearing load (pounds per square inch) and V equals shaft velocity (feet per minute).

From the foregoing, it will be appreciated that the objects of this invention have been obtained. A low density bearing of excellent properties can now be fabricated by low pressure compaction and without the need for hazardous metal oxide reduction techniques.

I claim:

1. A method of producing porous metal objects by the sintering of powdered metal which comprises blending a powdered metal selected from the group consisting of aluminum, aluminum and metal alloying mixture and aluminum base alloys with a fatty acid having at least about 12 carbon atoms and a wax having a melting point not exceeding about 150° C. so as to form a mixture of metal powder, fatty acid, and wax, compacting the mixture into a green compact having a desired shape, substantially removing all the fatty acid and wax from the green compact prior to sintering of the compact, and after such removal heating said compact to sintering temperature, said removal being effected by placing the green compact in a chamber and evacuating the chamber to a pressure less than atmospheric, heating the green compact to a temperature not exceeding about 345° C. to volatize substantially all the fatty acid and wax components of the compact, and bleeding a gas into the chamber in direct contact with the compact so as to sweep the volatilized fatty acid and wax from the chamber prior to heating said compact to sintering temperature.

2. The method of claim **1** wherein the fatty acid has from about 12 to 22 carbon atoms and wherein the wax comprises an ester produced by the interreaction of a fatty acid and a compound containing at least one active hydrogen atom.

3. The method of claim **1** wherein the mixture of powdered metal, fatty acid and wax contains from about 0.1 to 1 weight percent of wax and from about 0.2 to 2 weight percent of fatty acid.

4. The method of claim **1** wherein the chamber is at a partial pressure of from about 200 to 3500 microns during heating of the green compact to effect removal of said fatty acid and wax.

5. The method of claim **1** wherein the fatty acid is selected from the group consisting of lauric acid, palmitic acid, stearic acid, oleic acid and mixtures of the foregoing.

6. The method of claim **1** for producing a bearing which comprises forming a mixture comprising a powdered metal selected from the group consisting of aluminum, aluminum and metal alloying mixture, and aluminum base alloys, a fatty acid having from about 12 to 22 carbon atoms and a wax having a melting point not exceeding about 150° C., said mixture containing about 75 to 95 weight percent of aluminum metal, about 0.1 to 1 weight percent of wax and from about 0.2 to 2 weight percent of fatty acid, compacting said mixture and placing the green compact in a chamber, evacuating said chamber to a partial pressure of from about 200 to 3500 microns and heating the green compact to a maximum temperature of about 345° C., and simultaneous with said heating bleeding an inert gas into the chamber in direct contact with the compact.

7. The method of claim **6** wherein the fatty acid is selected from the group consisting of lauric acid, palmitic acid, stearic acid, oleic acid and mixtures of the foregoing, and wherein the wax is selected from the group consisting of an ester produced by the interreaction of a fatty acid having from 12 to 34 carbon atoms with an alcohol having a maximum of two hydroxyl groups, amides of fatty acids having from 12 to 34 carbon atoms, and mixtures of the foregoing.

8. The method of claim **6** wherein the fatty acid is stearic acid.

9. The method of claim **6** wherein the wax is ethylene distearamide.

10. An aluminum bearing produced in accordance with claim **1** wherein the mixture comprises a minimum of about 75 weight percent of aluminum, from about 0.1 to 1 weight percent of wax and from about 0.2 to 2 weight percent of a fatty acid having from about 12 to 22 carbon atoms, wherein said mixture is compacted in a briquette die under a pressure of about 2 to 10 tons per square inch to form a green briquette, wherein the fatty acid and wax components of the green briquette are removed prior to sintering of the green briquette by placing the briquette into a chamber and evacuating the chamber to a partial pressure in the range of about 200 to 3500 microns while simultaneously heating the briquette to a temperature in the range of about 200 to 345° C. and bleeding an inert gas into the chamber in direct contact with the briquette so as to sweep the volatilized fatty acid and wax from the chamber, and finally sintering the briquette by heating it to a temperature of at least about 500° C.

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CARL D. QUARFORTH, *Primary Examiner.*

A. J. STEINER, *Assistant Examiner*