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**AIR SINTERING OF ALUMINUM
POWDER COMPACTS**
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4 Claims

ABSTRACT OF THE DISCLOSURE

Aluminum metal powder compacts are so made that they can be sintered in ambient atmospheric air with resulting outstanding physical properties. The controls which make this possible include the use of a controlled amount of magnesium or zinc, or both, in the powder mixture, compaction to 92 to 97% of theoretical density, the absence of any component in the powder mixture which would preclude direct metal-to-metal interface contact between the metal particles or volatilized during heating up to sintering temperature, the use of a normally liquid oil as a die wall lubricant, control of the heating-up rate to sintering temperature, and use of a relatively short sintering period.

This application is a continuation-in-part of my application Ser. No. 53,187, filed July 8, 1970, now abandoned.

This invention relates to the sintering of aluminum powder compacts and, more particularly, to the sintering of such compacts in the ambient atmospheric air.

The literature and patents on the production of aluminum powder metallurgy parts are replete with instructions on how to establish and maintain a non-oxidizing atmosphere within and about such parts during their heating-up to, and maintenance at, sintering temperature. The reason for such precautions is the propensity of aluminum powder to oxidize at elevated temperatures and the known fact that uncontrolled oxidation of the particles of an aluminum powder compact leads to catastrophic failure either during or after sintering.

The article by Cremer and Cordiano in the Transactions of the AIMME, volume 152 (1943), pages 152-165, contains the most complete discussion of sintering aluminum and aluminum-base powder mixtures in air. Although the authors reported successful air-sintering of aluminum and aluminum-copper powders which had been subjected to green compaction pressures of 40 to 60 tons per square inch and then sintered for 30 minutes, they emphasized that adequate results could not be obtained if the aluminum powder mix contained magnesium either in elemental form or as a magnesium-aluminum alloy powder.

I have now discovered that aluminum powder compacts of high strength, high ductility and controlled dimensions can be produced by powder metallurgy techniques while sintering the compacts in the ambient air provided certain critical conditions are maintained. This result is achieved pursuant to the present invention by

(a) intimately admixing with the aluminum powder, and in direct metal-to-metal interface contact therewith, at least one metal powder of the group consisting of magnesium and zinc, the magnesium being present in amount of at least 0.1% and the zinc being present in amount of at least 1% by weight of the aluminum so as to form with the aluminum at pre-sintering temperatures within the range of about 400-475° C. a protective layer of a low melting point eutectic of said metal and the aluminum,

(b) lubricating the walls of a powder metallurgy compaction die with a normally liquid lubricating oil,

(c) compacting the resulting metal powder in said lubricated die to a density ranging between about 92 to 97% of theoretical density,

(d) heating-up the resulting compact to sintering temperature in ambient atmospheric air at a rate of at least about 40° C. per minute, and

(e) holding the heated compact at sintering temperature for a maximum of about 10 minutes.

The method of the invention is applicable to air-sintering of virtually all grades of aluminum powder, the only limitation being that the finer the powder the poorer its flow rate for discharge into a die under commercial operating conditions. For example Alcoa's aluminum powder No. 1220 (9.7% minus 325 mesh Tyler), No. 120 (35.6% minus 325 mesh) and No. 123 (89% minus 325 mesh) have been used successfully with the conditional qualification, as mentioned previously, that the coarser powders had better flow characteristics. Other added metals, such as up to about 10% by weight of copper, up to about 3% by weight of magnesium and up to about 10% by weight of zinc were added advantageously as minus 325 mesh powders which were uniformly admixed with the aluminum powder.

In order to permit sintering of air, I have found that conditions must be such as to activate the sintering and to protect the aluminum particles against oxidation while they are being brought up to sintering temperature. In connection with activation of the sintering, I have found that the particles must be free from any material, such as conventional powder metallurgy internal lubricant, which would interpose a sinter-retardant layer or residue between the particles and thus detract from their direct metal-to-metal interface during heating-up and sintering of the compact. In fact, I have found it advantageous to the development of ultimate maximum desirable physical properties of the air-sintered final product that it not contain at any time any non-metallic lubricant or similar diluent or interferent with direct contacts between the metal powder particles.

The non-oxidizability of the green compacts in the practice of the invention is further enhanced by the presence in the aluminum powder of a small amount of another metal powder capable of forming a relatively low melting point eutectic with the aluminum. Thus, contrary to the teaching of Cremer et al., supra, I have found that a metal powder such as magnesium in amount of at least about 0.1% by weight or zinc in amount of at least about 1% by weight, and mixtures thereof, whether added as the individual metal powders or as an alloy of the two or in individual alloyage with aluminum or with another metal at least compatible with, or advantageously an additional component of, the aluminum-base product is a necessary component of the powder mix in accordance with the invention.

The primary effect of small additions of magnesium or zinc, or both, is to protect the aluminum powder against oxidation during heating-up to sintering temperature. It appears that oxidation of the aluminum particles does not take place to an objectionable extent until its temperature in ambient air reaches about 400° C. Sintering of commercial grade aluminum powder of conventional size for powder metallurgy takes place at about 650° C., thus leaving a temperature spread of about 250° C. during heating-up when oxidation can take place with adverse results. By avoiding the presence of internal lubricants or the like, sintering can be activated to the point where it takes place close to 600° C. and thus reduce to about 200° C. the heating-up temperature spread during which deleterious oxidation can occur. However, by adding the aforemen-

tioned small amount of magnesium or zinc, or both, a low melting eutectic of the magnesium or zinc with the aluminum forms in the neighborhood of 375°–450° C. and provides a protective film about the aluminum particles to prevent their oxidation. Under these conditions there is at most about a 50° C. spread within the heating-up temperature range during which oxidation can take place, and this spread is in the lower part of the oxidation temperature range where oxidation takes place more slowly.

The addition of magnesium or zinc to the aluminum causes expansion in the low melting eutectic roughly in proportion to the amount which has been added. Magnesium produces approximately ten times as much expansion as zinc, and the effect of the two metals, when both are used, is additive. As mentioned previously, either about 0.1% by weight of magnesium or about 1% by weight of zinc, or the equivalent of the two, is required to form a sufficient amount of low melting point eutectic to protect the aluminum powder against oxidation in the ambient atmosphere while it is being raised to sintering temperatures. Amounts of magnesium or zinc up to about 0.3% by weight of magnesium and up to about 3% by weight of zinc, or the cumulative equivalent amount of both metals, cause increasing expansion of the compact as it is brought up to sintering temperature but do not cause more than about 0.2% expansion of the compact. Up to this amount of expansion, the expansion does not open up the compact sufficiently to overcome the anti-oxidation protective effect of the low melting point eutectic. Above about 0.3% by weight and up to a permissible maximum of about 3% by weight of magnesium, or above about 3% by weight and up to a permissible maximum of about 10% by weight of zinc, or the cumulative equivalent amounts of both, the addition of these metals increases expansion of the compact significantly above 0.2% and appears to open up the compact to oxidation by the ambient atmosphere unless this expansion is compensated for by green compaction pressure. Thus, a further essential feature of my novel method of making possible air-sintering of aluminum is the use of sufficient compaction pressure to obtain a green density in the compact of between about 92 to 97% of theoretical density of the compacted material. This high green density contributes to the low interconnective porosity of the compact and assists in preventing internal oxidation of the compact during heating-up and sintering in ambient air.

The aforementioned high degree of compaction of unusually fine metal powder, without the use of an internal lubricant, depends in turn, pursuant to the invention, on the use of a normally liquid lubricating oil, applied by spraying, painting, swabbing, or the like, as a die wall lubricant during compaction. I have attempted to obtain the aforementioned characteristics in the green compacts with conventional die wall lubricants which are not normally liquid, including waxes, greases and soaps, as such or carried by solvents, and have been unable to obtain satisfactory air-sintered aluminum compacts even when using all of the previously discussed parameters for successful air-sintering at their optimum values. Hydrocarbon oils, particularly when fortified by flake graphite, are useful in the practice of the present invention, although I presently prefer silicon oils such as the methyl alkyl polysiloxanes having molecular weights ranging from 1,500 to 10,000. Such normally liquid materials, used as die wall lubricants, make possible the attainment, by high compaction pressure, of the necessary non-communicating internal porosity which precludes internal oxidation when the pressed green compacts are sintered in ambient atmospheric air pursuant to the invention. The alkyl groups in the preferred polysiloxanes include ethyl, propyl, butyl, amyl, hexyl, octyl, decyl, dodecyl and tetradecyl radicals which form long chains of polysiloxanes having linear organic radicals ranging from 1 to 40 carbons in length. A full description of these polysiloxanes appears in The Transactions of the American Society of Lubrica-

tion Engineers, volume 9, pages 31–35 (1966), in an article by E. D. Brown, Jr., entitled "Methyl Alkyl Silicones, A New Class of Lubricants."

After the compacts have been prepared as described hereinbefore, they are heated to proper sintering temperature. This heating-up, like the sintering operation, is carried out in ambient atmospheric air in a furnace capable of bringing the compacts up to sintering temperature, as described previously, at a rate of at least about 40° C. per minute and preferably at a rate of about 100°–200° C. per minute. Such relatively fast heat-up rates are essential to permit the relatively short time at sintering temperature which I have found to be essential to successful air-sintering pursuant to the invention.

The sintering temperatures can be substantially those used conventionally in the art of producing aluminum powder metallurgy parts, to wit, within the range of about 565°–650° C. For any specific powder mix composition, finer particle size generally dictates lower sintering temperatures and coarser powders require higher sintering temperatures within this range. Time at sintering temperature, on the other hand, is more critical because I have found that objectionable oxidation of the compacts takes place when sintering in air is carried out for the conventional periods of 15 minutes or more at sintering temperature. This observation is also related to the rate of heating-up to sintering temperature because slow heat-up rates make it difficult to determine, with such a low slope of the heat-up rate curve, when the compact has actually reached a sintering temperature. Moreover, because of the slow flattening of such a curve as it approaches the ultimate sintering temperature, it is impossible to obtain a short sintering time at sintering temperature. Accordingly, by using a steeper slope of heat-up curve, such as that of a heat-up rate of at least about 40° C. per minute, and preferably at a rate of about 100°–200° C. per minute, the time at sinter temperature can be determined with sufficient accuracy to assure that this time does not exceed about 10 minutes, and preferably ranges between about 3 to 8 minutes, before being cooled by exposing it to the ambient atmosphere. For any specific metal powder composition, shorter sintering time is desirable for higher sintering temperature and vice versa. The important criterion, pursuant to my discovery, is that the sintering temperature should not closely approach the liquidus temperature for the powder alloy composition and that time at temperature should be only a small fraction of heretofore customary sintering time in order to obtain actual sintering without excessive oxidation and resulting loss of desirable physical properties of the as-sintered product.

The following examples are illustrative but not limitative of the practice of the present invention.

Example I

A commercially pure aluminum powder (purchased on the market as Alcoa's No. 123 aluminum powder), having a particle size of at least 90% minus 325 mesh (Tyler Standard) and an apparent density of 0.90 g./cc. together with 2% by weight of minus 325 mesh copper powder and 0.3% by weight of minus 325 mesh helium-atomized magnesium powder, were mechanically mixed for about 30 minutes. The resulting powder blend was charged to a standard tensile bar die the walls of which had been coated with a methyl alkyl polysiloxane lubricant purchased on the market as General Electric's Silicone Oil No. SF 1147. The die dimensions were such as to form a tensile test bar 2 inches long, ¼ inch wide and ⅛ inch thick. The compaction pressure was sufficient to compress the loose powder charge to 98% of its theoretical density. The ejected compact showed no surface scoring, laminations or cracks, and each of a number of such tensile test bars had a green strength in excess of 5,000 p.s.i.

These green test bars were placed in pairs in low-carbon steel boats which were carried on a conveyor through a 4 inch wide belt furnace 3½ feet long and having an

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ambient air atmosphere maintained at a temperature of about 595° C. in a zone about 18 inches long. The belt travelled through the furnace at a rate of 2 inches per minute to provide a sintering time of about 7 minutes after a heat-up rate of about 40° C. per minute. In order to insure stability of these sintering conditions, dummy boats were sent through the furnace both before and after the test bars to be sintered.

The resulting test bars had a lustrous surface, exhibited a growth in length of less than 0.2%, and reached an average bend-to-furnace angle of about 135°. The fractures were bright and clean with no evidence of internal oxidation below their surfaces. The ultimate tensile strength of the bars ranged between about 20,000 and 25,000 p.s.i. in the as-sintered condition and averaged about 4% elongation. The sample responded to artificial aging with resulting increase in Rockwell H hardness from 75-85 in the as-sintered condition to 80-91 in the aged condition.

Example II

A powder mix was prepared and formed into green test bars as described in Example I except that the aluminum powder was that purchased on the market as Alcoa's No. 120 aluminum powder having a particle size of about 35% by weight minus 325 mesh and an apparent density of 1.119 g./cc. These green test bars were placed in pairs in low carbon steel boats which were carried on a belt through a conventional hump furnace having an ambient air atmosphere maintained at a temperature of about 630° C. The belt speed of 24 inches per minute provided a heat-up rate of 143° C. per minute and the compacts were at their ultimate sintering temperature for 3.8 minutes. The sintered bars had an average ultimate tensile strength of 23,500 p.s.i., a yield strength of 16,600 p.s.i., an elongation of 5%, and expanded only 0.2% in dimensions over the green compact dimensions.

The air-sintering process of the invention makes possible the production of strong, ductile aluminum powder parts without distortion, such as warping, and with such a small degree of growth or expansion, i.e. dimensional stability, that no subsequent coining, machining or other sizing operation is required.

I claim:

1. The method of producing a high strength and ductile air-sintered compact of controlled dimensions and consisting essentially of aluminum powder wherein internal oxidation of the compact is prevented by

(a) intimately admixing with the aluminum powder, and in direct metal-to-metal interface contact therewith, at least one metal powder of the group consisting of magnesium and zinc, and the magnesium being present in amount of at least 0.1% and the zinc

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being present in amount of at least 1% by weight of the aluminum so as to form with the aluminum at pre-sintered temperatures within the range of about 375°-450° C. a protective layer of a low melting point eutectic of said metal and the aluminum,

- (b) lubricating the walls of a powder metallurgy compaction die with a normally liquid lubricating oil, (c) compacting the resulting metal powder in said lubricated die to a density ranging between about 92 to 97% of theoretical density, (d) heating-up the resulting compact to sintering temperature in ambient atmospheric air at a rate of at least about 40° C. per minute, and (e) holding the heated compact at sintering temperature for a maximum of about 10 minutes.

2. The method according to claim 1 in which the green compact is heated to sintering temperature at a rate of about 100°-200° C. per minute and is held at sintering temperature for a period of about 5 to 8 minutes and is then cooled in the ambient atmosphere.

3. The method according to claim 1 in which the sintering temperature does not significantly exceed the solidus for the alloy composition of the metal powder mixture.

4. The method according to claim 1 in which the lubricating oil is a methyl alkyl polysiloxane having a molecular weight within the range of about 1,500 to 10,000.

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