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3,544,392

**PROCESS FOR MAKING HIGH QUALITY HOT-
WORKED PRODUCTS FROM ALUMINUM
BASE ALLOY POWDERS**

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14 Claims

ABSTRACT OF THE DISCLOSURE

Production of articles of improved ultrasonic quality made of aluminous metal powder by compacting powder to from 60% to less than 100% of theoretical density, degassing the resulting compact by heating without sintering at 800 to 1125° F. in a substantially dry inert atmosphere substantially free of hydrogen and then substantially immediately compressing the heated compact to substantially 100% of its theoretical density before hot working.

BACKGROUND OF THE INVENTION

This invention involves production of aluminous metal powder alloy articles. More particularly, it involves a method for use of powder metallurgy techniques to produce from aluminous metal powder a substantially degassed shaped article of improved ultrasonic quality. The term aluminous metal as used herein refers to both aluminum and the alloys in which this element predominates. It also is intended to cover mixtures of aluminous powdered metals in which aluminum or aluminum base alloy powder is the major component. The invention herein described was made in the course of or under a contract or subcontract thereunder with the Department of the Army.

It is known, for example, as taught in Ennor and Lyle U.S. Pat. 2,809,891, to compact and heat aluminous metal powder in a protective atmosphere for ¼ to 36 hours at an elevated temperature of between 840° and below the 1220° F. liquidus temperature of aluminum to a density of less than 100% of theoretical, to heat the resulting compact to a hot working temperature and then to hot work the final compact, for example, by transferring to an extrusion die. Theoretical density is the density of a completely consolidated hot worked article which has no substantial porosity. Towner and Lyle, in U.S. Pats. Nos. 2,966,731-5 and 2,963,780, disclose initially heating aluminum base alloy powder at 700-900° F., compacting the powder at 200 to 150,000 p.s.i. for from a minute to several hours, heating the compacted powder at 700-900° F. (except for an alloy containing 3% chromium, molybdenum, tungsten, titanium, zirconium, or manganese, which may be heated to 1150° F.), and then compressing the heated powder and finally hot working it by extruding, rolling, or forging. MacDonald and Ransley, in Symposium on Powder Metallurgy (England, 1954) at p. 245 of a paper entitled, "Preparation of High Modulus Aluminum Alloys by Powder Metallurgy," disclose sintering a nickel aluminum alloy powder in highly purified hydrogen at about 1137° F. for an hour and then hot pressing the sintered powder to close to 100% of its theoretical density. Blisters, discontinuities or voids are sometimes noticeable in articles produced by such fabricating procedures, especially after the heating which is used in the extrusion or shaping step or in subsequent solution heat treatment, artificial aging or annealing in specific instances, thus making them less acceptable under current requirements for substantial freedom from defects

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in certain airframe components which must undergo conditions of high stress.

OUTLINE OF THE INVENTION

It is accordingly an object of this invention to provide a process for preparation of aluminous metal powder articles of improved structural soundness. It is a further object of this invention to provide a method for treatment of aluminous metal powder such that the articles produced are substantially free from defects such as blisters, discontinuities and voids. Further objects of this invention will be apparent from the description and claims which follow.

Our invention is predicated upon the discovery that when aluminous metal powder is compacted to above 60% but less than 100% of theoretical density, preferably 65-90%, and then heated without sintering in a flowing, substantially dry inert atmosphere which is substantially free of hydrogen at from about 800 to about 1125° F. and then substantially immediately compressed to above 90% of theoretical density, that is substantially 100% of theoretical density and hot worked into a shaped article (for example, by hot pressing and then extruding), the presence of blisters, discontinuities and voids in the hot worked product can be minimized or avoided completely, and an improved ultrasonic rating imparted to the article. By improved ultrasonic rating or quality we mean one which is at least as high as the Class A standard (we use A+ herein to indicate this improved ultrasonic rating) of the Society for Nondestructive Testing (SNT) Airframe Subcommittee Report No. 1 (Revised), Feb. 28, 1964, pp. 366, 367 and 368, entitled, "Recommended Ultrasonic Acceptance Standards for Airframe Aluminum Alloy Plate, Extrusion and Forgings." This is a standard ultrasonic inspection test for checking against discontinuities which are not generally visible to the naked eye. Some evidence of improved ultrasonic rating is also to be found by visual inspection for absence of blistering and cracking. We have found that aluminous metal powder articles produced by our method even after the solution heat treatment and artificial aging required for some end uses and as described in Sprowls et al. U.S. Pat. No. 3,198,676 retain the improved or A+ ultrasonic rating or quality.

In stating that the inert atmosphere used in the heating step must be substantially dry, we mean an inert atmosphere which has a dew point of -40° F. or lower.

According to our process the heating step which follows the initial compacting step may take from about 1 to about 36 hours. The preferred time is from 12-24 hours. In the heating step it is essential that the temperature be high enough to be of help in the compression step which follows substantially immediately but not high enough to result in sintering. Thus, it should be above about 800° F. but below the fusion point of aluminum and not higher than about 1125° F.

While we do not wish to be bound by any theory, it appears that most aluminous metal powder contains at least some hydrogen (possibly formed from water present) and water entrapped therein which may be detrimental to the preferred substantially blister-free, void-free and discontinuity-free condition. Furthermore, the heating and compacting procedures heretofore known apparently tend to cause additional oxidation inside any pores which may exist, thus possibly helping to keep the aluminous metal powder articles from achieving the ultrasonic rating sometimes required. Therefore, as mentioned above, in our process we use a substantially dry and substantially hydrogen-free inert atmosphere in the heating step which substantially immediately precedes our compressing step. The inert atmosphere must also be

flowing in order to carry away hydrogen and water and other gases or vapors evolved during the heating. Examples of suitable substantially hydrogen-free, flowing, substantially dry inert atmospheres includes a vacuum, nitrogen, helium and argon. One reason for the requirement according to our invention that the inert atmosphere be substantially hydrogen-free is that hydrogen is one of the gases believed to cause some of the difficulties sometimes encountered by way of occasional blisters, discontinuities and voids in compacted aluminous metal powder and articles formed therefrom.

Controlling the extent of compacting of the powder to from about 60 to less than 100%, preferably 65-90, of theoretical density, in the first compacting step, and compressing to substantially 100% of theoretical density substantially immediately after the intermediate heating step further assist in eliminating undesirable voids and defects. Best results were obtained when compacting in the first step at 72-87% of theoretical density. The intermediate heating step between the initial compacting step and the final compression prior to hot working also permits bringing of the compacted powder to the temperature desired for the hot pressing step.

It may be desirable to use a heated compression chamber during the final compression step prior to hot working. The temperature during this compression and in the subsequent hot working generally ranges from around 500-900° F., preferably from about 600 to about 700° F. Ordinarily at least 40,000 p.s.i. pressure is desirable to obtain substantially 100% of theoretical density desired with absence of any substantial amount of porosity. The hot working may be by extrusion, forging, rolling or the like, our preferred method being extrusion.

The aluminous metal powders which are adapted to be compacted, heated, compressed and hot worked according to our process may be of the milled or of the atomized type. Powder prepared by air atomization and collection in air is preferred to eliminate as much undesirable moisture as possible. Atomization and collection procedures such as those described in the aforementioned Towner and Lyle U.S. Pats. Nos. 2,966,731-5 and 2,963,780 may be used. A wide variation in particle size is permissible, but the particles are preferably not larger than will pass through a 100-mesh screen. It is preferred to utilize powders of a very fine mesh size, preferably of from 5 to 60 microns mass median diameter (MMD) as measured by a Sharples micromerograph. Best results are obtained when the particles are from 18 to 54 microns in size.

The metal powder used in our process may consist of aluminum of commercial purity, for example, 99%, up to the highest purity obtainable, or it may consist of particles of aluminum base alloys, or it may be made up of a mixture of particles of aluminum and the desired alloying elements. The elements which may be thus associated with aluminum in either alloyed or elemental form are those commonly employed in the aluminum base alloy art, such as, copper, magnesium, silicon, zinc, manganese, iron and certain high melting point elements. They may be present in the quantity normally used in the solid metal alloys, for example, up to 10% copper, up to 15% silicon, up to 13% zinc, up to 10% magnesium. Such high melting point elements as nickel, chromium,

titanium, boron, and zirconium may also be employed in amounts up to 0.75% each. Especially when the aluminous metal powder is an alloy, as indicated above, it may be desirable to subject the compact or subsequent product to a specific thermal treatment with or without a subsequent precipitation hardening treatment.

For our initial compacting step any temperature below which melting begins is generally satisfactory. The pressure range is not critical, it being sufficient to use a pressure which will give the desired amount of compacting to eliminate too much porosity and yet to provide a compact of the desired strength which will withstand the conditions encountered in the subsequent heating and hot working steps. Room temperature (65-90° F.) is usually satisfactory. Higher temperatures, of course, require lower pressures to obtain the same desired density. As indicated above, the heating temperature for the intermediate heating step which follows the initial compacting step must be below that at which sintering of the powder occurs, and the temperature during the subsequent compression and hot working steps must also be below the fusion point.

The table which follows shows the characteristic ultrasonic rating as well as visual inspection appearance for representative aluminous metal powder articles prepared according to our invention. Air atomized and air collected powder was used. A flowing atmosphere of argon (10 cu. ft./min.) substantially free of hydrogen and moisture was used during the intermediate heating step which preceded the hot working. For our initial compacting step, we used a compression chamber which had a smooth cylindrical portion at one end and an outward taper on the other end. A hydraulically operated dummy block moved in the cylindrical portion toward a blind die during a compacting stroke, stopping when the face of the dummy block reached the point where the taper began. The blind die was removed and force applied to the dummy block to push the compact out of the compacting die. The cylinder used was about six inches in diameter, and the tapered wall portion had about a 1° taper. The cylinder was lubricated with butyl stearate before the powder was charged into it.

Heating for the intermediate heating step prior to the final compression step was at the temperatures and for the times shown in the table. In the compression step which follows the intermediate heating step and prior to the hot working step, the samples were pressed against a blind die in an extrusion cylinder at substantially full press capacity of about 93,000 p.s.i. and at temperatures below the fusion point. For the hot working step, the blind die was replaced with a 2"-diameter extrusion die and the article then extruded at a temperature of about 650° F.

The test procedure used for determining the ultrasonic rating of the samples in the table which follows (except for Prior Art Samples 1 and 2, for which a visual rating was obtained) was substantially the same as the SNT method referred to above except that a combination of reference block and instrument settings was used which was capable of detecting much smaller discontinuities than the SNT Class A method, the ratio of discontinuity sizes being about 1 to 8.

TABLE

Sample	Powder size, MMD	Pressure in applied initial compacting p.s.i. step, thousands	Percent of theoretical density to which compacted in initial compacting step	Temperature during heating step prior to compressing step, ° F.	Time of heating step prior to compressing step, hours
Prior art 1 ^{1,11}	—100 mesh—	93	—	900	1
Prior art 2 ^{2,11}	—100 mesh—	93	—	1,000	1
1 ¹²	38μ	36	76	1,100	13.5
2 ¹³	35μ	87	80	900	8.5
3 ^{4,13}	37μ	92	85	860	11.25
4 ^{5,13}	41μ	102	84	900	18.5
5 ^{6,10,12}	27μ	110	77	860	12.25
6 ^{7,10,12}	31μ	90	83	850	19

See footnotes at end of table.

TABLE—Continued

Sample	Blistering (visual)				Cracking (visual)			
	Before SHT ⁹	After SHT ⁹	Before SHT ⁹ and AA ¹⁰	After SHT ⁹ and AA ¹⁰	Before SHT ⁹	After SHT ⁹	Before SHT ⁹ and AA ¹⁰	After SHT ⁹ and AA ¹⁰
Prior art 1				Yes	Yes		Yes	Yes
Prior art 2						Yes		Yes
1	No	No	No	No	No	No	No	No
2	No	No	No	No	No	No	No	No
3	No	No	No	No	No	No	No	No
4	No	No	No	No	No	No	No	No
5	No	No	No	No	No	No	No	No
6	No	No	No	No	No	No	No	No

Ultrasonic rating				
Sample	Before SHT ⁹	After SHT ⁹	Before SHT ⁹ and AA ¹⁰	After SHT ⁹ and AA ¹⁰
Prior art 1	Poor (visual)		Poor (visual)	
Prior art 2		Poor (visual)		Poor (visual)
1	A+	A+	A+	A+
2	A+	A+	A+	A+
3	A+	A+	A+	A+
4	A+	A+	A+	A+
5	A+	A+	A+	A+
6	A+	A+	A+	A+

¹ Prior art sample 1 contained in percent by weight 6.1 Zn, 2.9 Mg, 1.6 Cu, 0.24 Cr, 0.05 Ti, 0.5 Al₂O₃.

² Prior art sample 2 contained in percent by weight 9.8 Zn, 3.5 Mg, 1.0 Cu, 0.04 Ti, 0.46 Mn, 0.4 Al₂O₃.

³ Sample 1 contained in percent by weight 5.5 Zn, 2.5 Mg, 1.6 Cu, 0.25 Cr.

⁴ Sample 2 contained in percent by weight 10.9 Zn, 4.9 Mg, 2.0 Cu, 1.7 Mn.

⁵ Sample 3 contained in percent by weight 10.3 Zn, 2.9 Mg, 2.1 Cu, 1.8 Mn, 0.14 Cr.

⁶ Sample 4 contained in percent by weight 7.8 Zn, 3.5 Mg, 1.6 Cu, 0.6 Mn, 0.5 Cr, 0.5 each Fe, Co, Mo, W, 0.4 each Ti, V, 0.6 each Zr, Ni.

⁷ Sample 5 contained in percent by weight 9.8 Zn, 4.2 Mg, 4.4 Cu, 2.1 Mn, 1.6 Fe, 4.6 Ni.

⁸ Sample 6 contained in percent by weight 2.9 Zn, 14.6 Mg, 2.4 Cu.

⁹ Solution heat treatment 2 hours at 860° F.

¹⁰ Artificial aging or age hardening 24 hours at 250° F.

¹¹ Prior art samples 1 and 2 were air atomized, air collected, screened, compacted in a 6.38" diameter cylinder at 93,000 p.s.i. and cylinder temperature of 650° F., ejected, then heated in air 1 hour at 900–1,000° F., cooled and scalped to 6.25" diameter, reheated and extruded to 2" diameter rods.

¹² Samples 1–6 contained less than 1% by weight Al₂O₃.

From the foregoing data it is readily apparent that, by compacting aluminous metal powder to from about 60 to less than 100% of theoretical density, degassing the compact by heating in a substantially dry, flowing inert atmosphere substantially free of hydrogen and then substantially immediately compressing the heated compact to substantially 100% of theoretical density prior to hot working, we have substantially eliminated blisters, discontinuities and voids caused by entrapped hydrogen and/or other gases, and enable production of aluminous metal powder articles which have an improved ultrasonic rating.

While the invention has been described in terms of preferred embodiments, the claims appended hereto are intended to encompass all embodiments which fall within the spirit of the invention.

Having thus described our invention and certain embodiments thereof, we claim:

1. A process for the production of aluminous metal powder articles which comprises

- (1) compacting aluminous metal powder to from about 60° to less than 100° of theoretical density,
- (2) heating the resulting compact below the fusion point of the metal in a flowing inert atmosphere substantially free of hydrogen at from about 800° F. to about 1125° F., thereby minimizing formation of blisters, discontinuities and voids during subsequent heating,
- (3) then compressing said compact to above 90% of theoretical density, and
- (4) hot working said compact into a shaped article having an improved ultrasonic rating.

2. The process of claim 1 wherein the compacting of the powder is to 65 to 90% of theoretical density.

3. The process of claim 1 wherein the aluminous metal powder comprises air atomized and air collected powder screened to a particle size of from 5 to 60 microns mass

median diameter, and the heating step is performed in an argon atmosphere.

4. The process of claim 1 wherein the heating is for from about 12 to about 24 hours and the hot working is followed by solution heat treating.

5. The process of claim 1 wherein the hot working is followed by solution heat treating and artificial aging.

6. The process of claim 1 wherein the hot working comprises extrusion.

7. The process of claim 1 wherein the hot working is followed by solution heat treatment.

8. The process of claim 1 wherein the hot working is followed by solution heat treatment and then artificial aging.

9. The process of claim 1 wherein the aluminous metal powder comprises air atomized and air collected powder screened to a particle size of 18–54 microns mass median diameter and the hot working is followed by solution heat treatment and then artificial aging.

10. The process of claim 1 wherein the aluminous metal powder comprises an aluminum base alloy containing zinc and magnesium.

11. The process of claim 1 wherein the aluminous metal powder comprises an aluminum base alloy containing zinc, magnesium and copper.

12. A process for preparation of an aluminous metal powder shaped article which comprises the following steps in successive order:

- (1) atomizing a melt of an aluminous metal in air,
- (2) collecting the resulting aluminous metal powder in air,
- (3) screening said powder to a particle size of 18–54 microns mass median diameter,
- (4) compacting said powder to from 65–90° theoretical density,
- (5) heating the compacted powder below the fusion point of the metal at 800–1125° F. in a flowing inert

atmosphere substantially free of hydrogen for 8-24 hours,

(6) then hot pressing the heated compacted powder to above 90% of theoretical density, and then

(7) extruding the resulting hot pressed, heated compacted powder, thereby forming a shaped article which has an improved ultrasonic rating when in solution heat treated condition.

13. The process of claim 12 wherein the aluminous metal powder comprises an aluminum base alloy containing zinc and magnesium.

14. The process of claim 12 wherein the aluminous metal powder comprises an aluminum base alloy containing zinc, magnesium and copper.

References Cited

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,544,392 Dated December 1, 1970

Inventor(s) John P. Lyle, Jr. and Raymond J. Towner

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 5, Line 60, change "60°" to --60%-- and "100°"
to --100%--

Col. 6, Line 72, change "90°" to --90%--

SIGNED AND
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Attest:

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