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2,947,068

ALUMINUM BASE POWDER PRODUCTS

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The invention described herein may be manufactured and used by or for the Government of the United States of America without the payment of any royalties thereon or therefor.

This invention relates to aluminum base metallurgical compositions and products produced therefrom, and to a method for the production of such products.

More particularly, the present invention is concerned with metallurgical powder compositions comprising substantially non-oxidized aluminum or alloys thereof and oxygen bearing compounds of aluminum, and structural material produced therefrom through the utilization of suitable methods and techniques of powder metallurgy.

This application is the continuation-in-part of my application, Serial No. 496,087, filed March 22, 1955 for "Aluminum Base Powder Products," now abandoned.

Heretofore, the prior art has considered that the presence of substantial quantities of metal oxides in metal powder compositions was detrimental to the useful properties of the material produced from such compositions. For this reason in processes employing powdered aluminum or alloys thereof, the presence of aluminum oxide has been minimized or avoided wherever possible. Contrary to the teachings of the prior art, however, I have found that in such processes the amount and distribution of the oxygen bearing compounds of aluminum are properly controlled, such oxygen bearing compounds of aluminum may not only be present in substantial quantities but act to enhance the strength and hardness of the products formed of such aluminum and aluminum alloy powder compositions.

For example, structural material possessing unusual and desirable properties may be produced by suitable powder metallurgical processes from compositions of substantially non-oxidized powders of aluminum or alloys of aluminum and aluminum oxide. The forms produced therefrom and in accordance with this invention are characterized by relatively light weight and greatly improved strength and hardness at elevated temperatures up to about 1200° F.

Accordingly, it is a principal object of this invention to provide compact substantially non-porous aluminum base structural material and forms therefrom characterized by light weight and greatly improved strength and hardness at temperatures up to 1200° F.

Another object of the invention was to provide new and improved powder metallurgical compositions of substantially non-oxidized aluminum or alloys thereof and oxygen bearing compounds of aluminum.

It is a further object of the invention to provide a method of producing such compact, substantially non-porous structural material and forms therefrom from the before mentioned metallurgical compositions by utilization of suitable techniques of powder metallurgy.

Other objects and the attendant advantages of the invention will be obvious to those skilled in the art from the more detailed description set forth below, it being understood that such description as given by way of

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illustration and explanation only and not by way of limitation, since various changes therein may be made by those skilled in the art without departing from the spirit and scope of the invention.

Broadly stated, the foregoing objects are accomplished by means of a process essentially comprising the following steps:

(1) A powder composition is first prepared by mixing a metallic base of substantially non-oxidized aluminum or aluminum alloy powders and finely divided oxygen bearing compounds of aluminum. The powder composition is preferably milled for fifteen minutes to one hour in a ball mill with sufficient wetting agent such as xylol to form a soft paste.

(2) The powder composition prepared as stated above is then presintered at a temperature of from about 800° F. to about 1100° F., but not above the melting point of aluminum, under non-oxidizing conditions to remove the lubricants therefrom.

(3) The presintered powder composition is then cooled to room temperature before exposure to air.

(4) The powder compositions prepared in accordance with the above process are then subjected to suitable techniques of powder metallurgy to produce the desired forms.

The process, as briefly set forth above, is hereinafter described with particular reference to producing compacts from a powder composition comprising approximately 90% low lubricant, substantially unoxidized, flake aluminum powder and about 10% finely divided aluminum oxide. However, it will be appreciated that the invention is not limited to such specific composition or to the production of such compacts, and that other suitable well known powder metallurgical techniques may be used to produce larger or more complicated shapes from suitable powder compositions produced utilizing the process described in steps 1 through 3 set forth above.

Regardless of the final form, the structural material and bodies therefrom produced in accordance with the invention are characterized as relatively light weight and compact substantially non-porous bodies of unusually high strength and hardness at elevated temperatures up to about 1200° F., as distinguished from conventional porous ceramics having relatively low physical strength such as are produced by the process disclosed in the United States Patent No. 2,568,157 to J. M. Lepp et al.

The following specific example describing the preparation of a metallurgical composition in accordance with the invention and manufacture of a form therefrom and the hot hardness testing of the same is given for purposes of illustration only:

EXAMPLE

A dry powder composition was prepared from 90% by weight low lubricant, substantially unoxidized flake aluminum powder and 10% by weight of finely divided aluminum oxide powder. The aluminum powder was of such size as to pass a 325 mesh screen and the aluminum oxide powder had an average particle size of 12.5 μ . The above prepared powder composition was milled for about one hour with sufficient xylol to form a soft paste. The milled charge was then placed in an aluminum boat and presintered for one hour at about 800° F. under inert (argon) atmosphere. The presintered charge was then cooled to room temperature before being exposed to air.

Compacts were made from the above described powder composition using a double action die and at a pressure of 40 tons per square inch. Pressures greater than this tended to cause welding of the compacts to the walls of the die and in some instances resulted in fracturing of the die as well as cracking of the specimen upon ejection from the die.

The compacts, prepared as above, were then sintered

at a temperature of about 1000 to 1050° F. for about one hour under an inert atmosphere (argon) followed by air cooling to room temperature.

It has been found that compacts which are harder at room temperature are usually harder at elevated temperatures. Accordingly, all compacts were repressed after sintering by a coining process and in the same die used for making the compact. It was noted in some instances that due to cold welding the pressed compacts were about as hard after pressing as after complete processing. It appears therefore that where cold welding is achieved during the pressing step, the sintering step may be omitted in some instances.

Hot hardness testing of the compacts as above prepared was chosen because of the well known correlation between hot hardness, creep and creep rupture strength. Compacts prepared in accordance with the invention were hot tested for hardness using a Rockwell Superficial Hardness Tester with a 15 kg. load and $\frac{1}{8}$ steel ball indenter (Rockwell W. Scale). The anvils were cylindrical stainless steel blocks 3" in height and $2\frac{1}{2}$ " in diameter with cylindrical cavities in the top. A larger anvil was used to support the stainless steel block and a $\frac{1}{4}$ " sheet of transit was interposed between the larger anvil and the stainless steel block. The stainless steel blocks were heated to testing temperature and a prepared compact specimen placed in the cavity therein. The resulting assembly was furnace-heated to testing temperature and this temperature maintained for a period of one hour. The assembly was then removed from the furnace and readings taken as rapidly as possible with the described Rockwell Superficial Hardness Tester and using the Rockwell W. Scale. Examples of hot hardness data thus obtained were recorded in Table I appearing below.

Table I
HOT HARDNESS OF ALUMINUM BASE POWDER PRODUCTS

Percent Additive to Aluminum Powder	Rockwell Superficial Hardness number, R _w ($\frac{1}{8}$ " dia. steel ball—15 kgm. load)					
	R _w as Pressed	R _w Sintered	R _w Coined	R _w at 1,000° F.	R _w at 1,100° F.	R _w at 1,150° F.
Percent (Pure Aluminum).....	88	89	93	66	50	23
5% Al ₂ O ₃	85	86	89	69	53	36
10% Al ₂ O ₃	84	84	87	74	55	57
25% Al ₂ O ₃	85	84	86	72	58	51

It is apparent from Table I that finely divided aluminum oxide added to substantially unoxidized aluminum or aluminum alloy flake powder greatly increases the hardness and strength of the products produced therefrom at elevated temperatures. Powder compositions containing from 2 to 25% aluminum oxide were tested and were found to give results superior to aluminum at elevated temperatures. Those compositions containing from 5 to 15% aluminum oxide produced the best characteristics at elevated temperatures. Increasing the percentage of aluminum oxide powder in the composition above 25% tends to reduce the ductility of the resulting material.

In addition to aluminum oxide other oxygen bearing compounds of aluminum may be employed as additives in the metallurgical compositions of the invention. Such oxygen bearing compounds are the aluminates of barium, beryllium, calcium, cobalt, iron, lithium, manganese, magnesium and nickel and aluminum borate. These compounds have been tested in the ranges of 2-25% of the total compositions in the same manner as described above and have been found to have a hot-hardness and a tensile strength in the sintered and coined state comparable to compositions employing aluminum oxide. Other physical properties however vary depending on the particular composition tested.

Flaked aluminum and aluminum powders of the size which will pass a 325 mesh screen have been found to give the best results when used in the aluminum powder compositions of the invention. Less finely divided aluminum powder resulted in compacts which were not as hard at elevated temperatures. Atomized aluminum and aluminum alloy powders may also be employed if desired. The oxygen bearing compound of aluminum additive should also be in the finely divided state, the most satisfactory additives are quite fine powders composed of particles ranging between 5 and 15 microns.

The alloys of aluminum which have been found most useful for the purposes of the invention are those alloys known to be resistant to high temperatures and known in the art as strengthening metals for aluminum and aluminum alloys. Examples of such alloys are alloys of aluminum with iron, molybdenum, vanadium, titanium, beryllium, iridium, thorium, tantalum, niobium, chromium, manganese, nickel, cobalt, tungsten, boron and the rare earth elements. It has been found that one or more of such strengthening metals may be added in finely divided forms directly to metallurgical powder compositions described herein comprising aluminum and aluminum oxide and may constitute up to 50% of the metallic base with excellent results.

The purpose of the presinter is to remove the lubricant incorporated into the powder composition during the mixing step. Any satisfactory lubricant such as xylol, steirates or alcohols may be employed for this purpose.

A study of presintering conditions shows that, in most instances, a satisfactory presinter is obtained after one hour at 800° F. in an argon atmosphere. However, a satisfactory presinter may also be obtained, depending upon the characteristics of the particular composition and the lubricant employed, after a period of time varying from $\frac{1}{2}$ up to about 3 hours at a temperature of from about 700° F. to about the melting point of aluminum in any inert or non-oxidizing atmosphere. The presintering step may take place in any inert atmosphere or non-oxidizing condition including a vacuum, and argon or helium atmospheres. Control of the atmosphere during the first or presintering step is most important since it is at that time that the purity of the powder would be destroyed most rapidly.

A study of pressing pressures for the various mechanical and working processing of the composition described herein showed that in most instances a satisfactory pressure was about 40 tons per square inch. Pressures ranging from 30 to 75 tons per square inch may be employed successfully where the base metal contains a substantial portion of metal other than aluminum, but pressures less than 40 tons per square inch tend to produce unsatisfactory compacts in some instances while higher pressures tend to cause welding of the aluminum to the walls of the die and eventually result in the fracturing of the die as well as cracking of the specimen upon ejection from the die. However, regardless of the pressure used, the temperature at which the mechanical working and processing of the composition and forms is carried out should not exceed the melting point of aluminum since, if the temperature goes substantially above that point the physical properties of the product will be reduced.

It is to be understood that the invention is not limited to the specifically described addition to aluminum flake powder. Atomized aluminum powder or atomized aluminum alloy powder or aluminum flake or aluminum alloy flake or other suitable finely divided forms of aluminum or aluminum alloy or mixtures of the foregoing, may be employed in the compositions of the invention.

The powder compositions prepared in accordance with steps 1-3 of the described process may be processed into bodies of desired shape and size by the powder metallurgical methods specifically described or by any other suitable well known method not specifically described herein. For example, depending on the characteristics

of the particular powder composition, cold pressing or hot pressing alone may be sufficient. Cold pressing may be combined with sintering or sintering with hot pressing, or a combination of the foregoing steps may be employed prior to mechanical deformation. Forging, rolling, extruding or casting may also be employed either immediately after presintering or after the sintering and pressing operation. Where sintering is found to be desirable, the composition should be pressed prior to sintering. When this procedure is followed, sintering may proceed in any desired atmosphere provided the sinter is not unduly prolonged as will be understood by those skilled in the art.

Obviously, many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed as new and desired to be secured by Letters Patent of the United States is:

1. A structural material formed by mechanically working a metallurgical composition consisting of a finely divided mixture of substantially unoxidized particles of a metallic base and 2 to 25 parts by weight of particles of an oxygen bearing compound of aluminum said metallic base selected from the group consisting of aluminum, alloys of aluminum, and mixtures of aluminum and alloys of aluminum with each other and with aluminum alloy strengthening metals selected from the group consisting of iron, molybdenum, vanadium, titanium, tantalum, niobium, chromium, manganese, nickel, cobalt, tungsten, boron, beryllium, thorium, iridium, and the rare earth elements wherein said strengthening metals comprise less than 50 percent of said metallic base, said oxygen bearing compound being selected from the group consisting of aluminum oxide, the aluminates of barium, beryllium, calcium, cobalt, iron, lithium, manganese, magnesium, and nickel, aluminum borate and mixtures thereof, said composition having been presintered under non-oxidizing conditions at a temperature between about 700° F. to about the melting point of aluminum.

2. The structural material of claim 1 wherein the oxygen bearing compound of aluminum is aluminum oxide.

3. The structural material recited in claim 1 wherein said composition has been presintered at about 800° F. for about one hour.

4. The structural material of claim 3 wherein said composition has been mechanically worked at a pressure of about 40 tons per sq. in.

5. The method of producing a structural material capable of unusual strength and hardness up to about 1200° F. by presintering to a temperature from about 700° F. to a temperature not above the melting point of aluminum a composition consisting of a finely divided mixture of substantially unoxidized particles of a metallic base and 2 to 25 parts by weight of particles of an oxygen bearing compound of aluminum said metallic base selected from the group consisting of aluminum, alloys of aluminum, and mixtures of aluminum and alloys of aluminum with each other and with aluminum alloy strengthening metals selected from the group consisting of iron, molybdenum, vanadium, titanium, tantalum, niobium, chromium, manganese, nickel, cobalt, tungsten, boron, beryllium, thorium, iridium and the rare earth elements wherein said strengthening metals comprise less than 50 percent of said metallic base, said oxygen bearing compound being selected from the group consisting of aluminum oxide, the aluminates of barium, beryllium, calcium, cobalt, iron, lithium, manganese, magnesium, and nickel, aluminum borate and mixtures thereof, and mechanically working said presintered composition to form the structural material.

6. The method of claim 5 wherein said composition is presintered at about 800° F. for about one hour.

7. The method of claim 6 wherein said presintered composition is mechanically worked under a pressure of about 40 tons per sq. in.

References Cited in the file of this patent

UNITED STATES PATENTS

2,568,157 Lepp et al. ----- Sept. 18, 1951

FOREIGN PATENTS

779,573 Great Britain ----- July 24, 1957