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PRODUCTION OF ALUMINUM POWDER

Rudolf F. A. Wolf, Hamburg, Germany, assignor to Norddeutsche Affinerie, Hamburg, Germany

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The present invention relates to an improved process for the production of aluminum powder which is particularly suited for the production of sintered bodies of high strength and hardness even at higher temperature ranges.

In order for aluminum powders to be suitable for the production of such sintered bodies they must possess the following qualities: an oxide content of about 6–12%, which is usually provided by oxidation of the aluminum particles, and is present in finely divided form; a poured weight of at least 0.7 g./cm.^{3} and a lubricant content below 0.3%.

Aluminum powders of this type, having the above-mentioned qualities, are already known and they have previously been prepared by treating aluminum foil scraps, aluminum granules and the like in hammer mills and/or ball mills in a plurality of steps until the desired degree of fineness is achieved. As the comminution progresses, such processes must be carried out under protective gases having a lowered oxygen content so as to avoid danger of explosions on one hand and on the other hand provide for the desired oxidation. Towards the end of the processes, the grinding is effected with only a small quantity of lubricant in order that a partial welding of the particles is obtained which provides the desired higher poured weight of the resulting powder.

The mode of production employed produces a marked lamellar particle structure. The desired aluminum oxide content in the aluminum powder was partly achieved by surface oxidation of the new surfaces on the aluminum particles produced during the grinding operations and partly by a similar oxidation of the intermediate ground products in air during long periods of storage which must be interposed between the grinding operations and which can be of the order of weeks or even months.

The procedure described not only is cumbersome and time consuming, but also requires large expenditures for apparatus and energy and furthermore is subject to the danger of explosions.

It is the object of the invention to provide an improved process for the production of an aluminum powder of the indicated qualities which avoids the aforementioned disadvantages and furthermore provides considerable advantage.

According to the process of the invention the comminution is not effected by hammering or grinding of solid aluminum, but rather by comminution of molten aluminum to the required final extreme degree of fineness by spraying the melt. Furthermore, the oxide formation according to the invention is obtained by treatment of the aluminum powder obtained by the spraying operation with hot water, rather than by the limited oxidation obtainable during the grinding procedure and intermediate storage periods. The hot water oxidation not only is extraordinarily rapid, but is easily controlled to produce the desired and predetermined degree of oxidation. The aluminum powder which has thus already been extensively oxidized is only then subjected to an after-treatment in a ball mill. As the aluminum powder is already substantially in the desired state of comminution and oxidation, the time and energy required for the treatment in the ball mill are relatively slight and there is no longer any danger of explosion.

The preparation of the aluminum powder employed in the process according to the invention is preferably carried out by atomizing molten aluminum and quenching the atomized aluminum with water by the process described in German application N 867 Ib/49 1. According to such process, the molten aluminum is atomized into a closed chamber by permitting a stream of molten aluminum to fall free from an outlet in a crucible and projecting an annular stream of gases or vapors employed as atomizing medium against such falling stream of aluminum. Preferably, the atomizing medium is blown against the falling aluminum stream in the form of a cone and at, at least, ultrasonic speed, care being taken that the point where atomization occurs is spaced sufficiently from the outlet for the atomizing medium, such as an annular slit, so that the metal particles are not thrown back against the mouth of the atomizing nozzle by the eddies produced. This can, for example, be achieved by having the outlet for the metal stream project sufficiently beyond the annular outlet for the atomizing medium. A single annular nozzle or a plurality of nozzles concentric with the outlet for the molten aluminum stream can be employed to blow the atomizing medium against the molten aluminum stream. Steam at 3 to 15, preferably 8 to 12, atmospheres gauge pressure is expediently used as the atomizing medium. Water is sprayed from all sides into the atomizing chamber to condense the steam employed as the atomizing medium. For example, in atomizing 300 kg. of aluminum per hour, 1200 kg. of steam at 10 atmospheres gauge pressure and 30 m.^{3} of water are required per hour. The temperature of the water and aluminum powder mixture withdrawn from the atomizing chamber is controlled depending upon the composition of the aluminum to below 100° C. and preferably to about 25 to 50° C. If the temperature of such mixture is too high, lively hydrogen evolution occurs. The higher the magnesium content in the aluminum powder produced, the lower the water temperature must be to prevent evolution of hydrogen.

The aqueous aluminum powder slurry leaving the atomizing apparatus is pressed out in normal filter apparatus, particularly filter presses with an end pressure of 5 atmospheres gauge pressure to reduce the moisture content of the powder to about 9% H_2O. The resulting crumbly filter cake is then pressed through a sieve with an 8 mm. mesh width and dropped on flat drying pans to a 30 mm. height. The pans are then placed in a drying cupboard, preferably with circulating air, and dried first at 30° C., then slowly raised to 40 to 45° C. and finally to 60° C.

Previously in technical usage all contact of finely divided aluminum powder with water was avoided. However, according to the invention it was found that alumi-

num powder, produced by atomizing molten aluminum and quenching the atomized aluminum with water, reacts with water at elevated temperature with the formation of an oxide coating on the individual particles and that the quantity of oxide formed can be controlled, as it depends upon the water temperature and the duration of the treatment. The oxidation can, for example, be interrupted simply by adding cold water when the desired degree of oxidation has been reached.

The oxidation of the aluminum powder with warm water can be effected upon the dried product produced in the manner described above, but preferably such oxidation can be effected in the aqueous slurry produced by the atomization procedure either by adjusting the quantity of cooling water supplied to the atomizing chamber to provide the necessary temperature for the oxidation, the oxidation being controlled by controlling the time the aluminum powder remains in contact with the water at temperatures causing oxidation or by carrying out the atomization under conditions wherein the aluminum powder slurry produced is at a temperature lower than the temperature required for the oxidation and subsequently heating such slurry to the temperature for the period required for the oxidation.

The powder thus obtained by the atomization and oxidation despite its high oxide content, nevertheless, still is not suited for the production of sintered products of high strength and hardness at elevated temperatures, as the oxide only lies upon the surface of the individual particles as a relatively thick coating and is not dispersely distributed. According to the invention it was found that such atomized and oxidized powder can be rendered suitable for the production of sintered bodies of the type described by giving it an aftertreatment in a ball mill or similarly operating apparatus with the addition of a small quantity of lubricant. This aftertreatment to a great extent causes the oxide to be kneaded into the metallic base of the particles and provides fine distribution between the oxide and metal which is recognized as necessary in an aluminum powder for the production of sintered bodies of the type described.

The milling treatment, both in its purpose as well as its manner of being carried out, is substantially different from the grinding processes employed by the prior art in the production of aluminum powder of the type described. It is only of short duration and essentially requires no energy for comminution. Furthermore, it does not necessarily serve for a further oxidation. It also does not need to be interrupted by long storage periods and is not subject to dangers of explosion. The treatment of the oxidized aluminum powder according to the invention in a ball mill is completely safe as the oxide coating already present protects the metal from ignition and explosion. Consequently, no protective gases or other protective measures are required.

The entire preparation of the aluminum powder from ingot to finished product can be effected in a few hours. The oxidation itself only requires a few minutes.

The following example illustrates an embodiment of the process according to the invention.

Example

300 kg. per hour of molten aluminum were atomized in a chamber with the aid of 1200 kg. per hour of steam at 10 atmospheres gauge pressure as atomizing medium and 30 m.³ per hour of cooling water were sprayed into the chamber to produce an aqueous aluminum powder slurry having a temperature of about 40° C. 80% of the aluminum powder in such slurry had a particle size in the range of 1–40μ. Such powder in the dry state had a poured weight of about 1 g./cm.³.

The slurry was then heated to a temperature of 85° C. for 3 minutes whereby an 8% oxide content was achieved in the aluminum powder. The oxidation was terminated by cooling the slurry to below 40° C. by addition of cold water. The resulting oxidized powder was then filtered off and carefully dried at low temperatures so that practically all water was removed below a temperature of 40° C.

The oxide content of the treated aluminum powder can easily be controlled by selecting a proper temperature and duration of the oxidizing treatment. Oxidation begins upon heating the slurry over 50° C. and, as indicated, it can be terminated by cooling below 40° C. For example, a 25% oxide content can be achieved by heating the slurry at 85% C. for 10 minutes. Preferably, for the purposes of the invention, the oxide content of the final aluminum powder is between about 6% and 12%.

The resulting oxidized aluminum power in which the individual aluminum particles were coated with a relatively thick oxide skin was mixed with 0.5% stearic acid (lubricant) and treated for 5 to 6 hours in a ball mill under exclusion of air. The quantity of stearic acid remaining in the milled product was below 0.3% in view of reactions and evaporation. When the treatment of the oxidized aluminum powder is carried out under access of air, its oxide content increases by about 2–3%. This increase in oxide content must be taken into account when a final desired oxide content is to be obtained by effecting a lower degree of oxidation during the preceding oxidation in heated water.

The aluminum powder obtained after the treatment in the ball mill is admirably suited for the production of sintered bodies of high strength and hardness even at higher temperatures and possesses all of the qualities indicated as necessary for such a powder, namely, in having an oxide content between 6% and 12% and over and a poured weight of between 0.7 and 0.8 g./cm.³ and being practically fat free.

Sintered bodies produced from such powder had the following characteristic values:

Tensile strength at room temperature	32–35 kg./mm.²
Elongation at room temperature	6–8%.
Hardness at room temperature	About 80 kg./mm.² Brinell.
Hardness at 250° C.	About 50 kg./mm.² Brinell.
Hardness at 350° C.	About 35 kg./mm.² Brinell.

I claim:

1. In a process for the production of an aluminum powder having an aluminum oxide content of at least 6% rendering it particularly suited for the production of sintered bodies of high tensile strength and hardness even at elevated temperatures, said powder having a poured weight of about 0.7–0.8 g./cc. and over and a lubricant content of less than 0.3%, the steps which comprise preparing an admixture of water with an aluminum powder obtained by the atomization of molten aluminum, subjecting said admixture to an elevated temperature sufficient to effect surface oxidation of the aluminum powder, cooling said admixture to a temperature below that at which such surface oxidation occurs after the oxide content of the aluminum powder has reached at least 6% to terminate such oxidation, drying the oxidized powder, admixing the dried powder with a relatively small quantity of lubricant and treating such admixture in a ball mill to knead the oxide into the metallic base of the particles to obtain an aluminum oxide containing aluminum powder having the required fine distribution between aluminum metal and oxide to render it suited for the production of sintered bodies of high tensile strength and hardness even at higher temperatures the quantity of lubricant admixed with the dried oxidized powder being such that after the treatment in the ball mill the milled powder has a lubricant content less than 0.3%.

2. The process of claim 1 in which the aluminum

oxide content of said aluminum powder is between 6% and 12%.

3. The process of claim 1 in which said treatment in the ball mill is carried out in 5 to 6 hours.

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