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FABRICATION OF SPHERICAL POWDERS
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8 Claims

ABSTRACT OF THE DISCLOSURE

The instant disclosure teaches a process for obtaining spherical metal powders of a predetermined size range by admixing metal particles with a particular binder in the presence of moisture to form agglomerated masses of metal particles and binder, coating the masses with a weld preventing material and heating the resultant material.

The present invention relates to an improved process for obtaining spherical metal particles.

Numerous processes have been used for obtaining rounded or spherical metal powders or metal shot. The disadvantages of these processes are many fold. In particular, it is difficult to obtain a closely controlled particle size. Conventional processes are characterized by a wide distribution of particle sizes.

Furthermore, conventional processes are frequently expensive and frequently do not obtain spherical or rounded particles. During atomizing, particles produced by conventional processes are often hollow or have crevices or will be elongated or tear-drop in shape.

In addition to the foregoing there are numerous other disadvantages of conventional processes, which disadvantages will appear in part from the ensuing specification.

For example, a typical art process is the production of shot where the metal flows through an orifice in a thin stream which is then dispersed with high velocity jets of air, steam, water or heated gases.

Alternatively, shot may be produced by pouring molten metal on a rotating surface and then cooling with hot water.

The above methods do not produce a closely controlled spherical size. The yield of round particles is often poor and the yields of desired size will vary.

An alternative method is to cut wire of a predetermined diameter into small pieces. These particles are then partially rounded in a disc mill. This method does insure close particle size; however, the particles are not truly spherical and the material is expensive.

Accordingly, it is a principal object of the present invention to provide a process for the fabrication of rounded or spherical metal powders.

It is a further object of the present invention to provide a process as aforesaid which can obtain a high yield of particles of a predetermined size.

It is a further object of the present invention to provide a process as aforesaid which is inexpensive and readily adaptable to a commercial operation.

Further objects and advantages of the present invention will appear hereinafter.

In accordance with the present invention it has now been found that the foregoing objects may be readily obtained. The process of the present invention comprises:

(A) Providing powdered metal particles, preferably copper or copper base alloys, preferably having a particle size below 100 mesh,

(B) Providing less than 10% by weight of a binder which volatilizes above 150° F. and 200° F. below the melting point of said metal particles,

(C) Admixing said metal particles and binder in the

presence of from 5 to 35% by weight of moisture to form agglomerated masses of metal particles and binder, and preferably separating the desired size range of said masses,

(D) Coating said masses with a weld preventing material,

(E) Heating said coated masses in a protective atmosphere to a temperature above the volatilization temperature of said binder, and

(F) Heating in a protective atmosphere above the melting point of said metal to form a plurality of coherent metal spheres.

In accordance with the present invention it has now been found that the foregoing process readily obtains a high yield of rounded or spherical particles of predetermined size. The foregoing process is characterized by its relative simplicity and by being inexpensive. Furthermore, the particles obtained in accordance with the present invention are solid and rounded, are obtained in the desired particle size in a high yield, and are generally not hollow and generally do not have crevices.

In general, the process of the present invention makes particles of closely controlled size in very high yields. The process of the present invention makes spherical particles of materials and alloys which are not normally made. The spherical particles may contain metallic additives which do not alloy and also non-metallic additives as well.

In addition, the particles prepared in accordance with the process of the present invention will have a very low oxygen content since melting is done under a protective atmosphere. Thus, for example, copper shot made in this way can be considered as OFHC copper.

The convenient obtaining of the foregoing advantages of the present invention will be readily apparent from a consideration of the ensuing specification.

The present invention may be readily employed with virtually any metallic material. It is preferred that the metallic particles be provided in powdered metal form having a particle size below 100 mesh. The pure metal or alloys may be used. In addition, mixtures of metals may be also conveniently employed or their reducible oxides. For example, it is preferred to use copper or copper base alloys. Others which may be used include aluminum or aluminum alloys, ferrous materials, noble metals, precious metals, titanium, magnesium, zinc, nickel, beryllium, solders, and so forth.

A wide variety of binder materials may be employed. The binder material may be liquid or solid and should be a binder for the metal being used. The binder should volatilize above 150° F. and should volatilize at least 200° F. below the melting point of the metal being used. The particular binder which is employed is not especially critical. For example, one may readily employ brazing fluxes compatible with the base material, waxes, e.g. polyethylene glycol, metal stearates, stearic acid, organic binders, gums, etc. The binder is provided in an amount of less than 10% by weight. The lower amount will vary depending on characteristics of the particles.

In general, the particular choice of the binder will be influenced by the metal particles being employed.

The binder material should volatilize within the foregoing temperature range. It is not necessary that the binder completely volatilize, but some residue may be left as long as the residue comes to the surface, i.e., as long as the binder leaves the agglomerated masses.

The binder and metal particles are admixed in the presence of from 5 to 35% by weight of moisture to form agglomerated masses of metal and binder. Any liquid may be employed as moisture. It is preferred to use water, but many others may be employed, for example, alcohols,

acetone, toluene, benzene, naphtha, carbon tetrachloride, trichlorethylene, etc.

The amount of moisture content used may vary widely within the above range depending upon the particular materials being employed.

The order of addition of moisture, binder and metal particles is not particularly critical. One could add the binder in a variety of forms, e.g., dry powder, or dissolved in the moisture. Alternatively, one may add the moisture and binder as a fine spray.

The constituents are admixed to form agglomerated masses of metal and binder. A moderate admixing is preferred in order to avoid the formation of a paste like mass. It has been found that the higher the moisture content, the faster the ball of agglomerated mass is formed. But the higher the moisture content the wider the size spread of the balls of agglomerated masses.

Subsequent to the formation of the agglomerated mass, it is preferred to screen or remove those masses of desired size. Naturally, the admixing is regulated so that the time of admixing is chosen to give the highest proportion of desired mass size. After the desired mass sizes are removed, the under or over size materials may be put back in the process and reworked.

The agglomerated masses or balls are then coated with any desired weld preventing material. The particular weld preventing material is naturally dependent upon the type of metal particles employed. For example, one may readily employ talc, lime, graphite, alumina, titanium dioxide, zirconium oxide, flour, non-reducible oxides, and mixtures thereof.

After the coating of the agglomerated masses, the coated masses are heated in two stages in a protective atmosphere. The atmosphere may be neutral on the reducing side. One may use a lean gas or a strongly reducing gas. For example, one may employ hydrogen, carbon monoxide, dissociated ammonia, nitrogen, argon, helium and so forth. Alternatively, a vacuum or partial vacuum may be used as a protective atmosphere.

The first stage is to heat to a temperature above the volatilization temperature of the binder in order to remove the binder from the agglomerated mass. Naturally, one should not heat above the melting point of the metal.

As stated hereinabove, the binder should be such as to completely volatilize or partially volatilize with the remainder coming to the surface, i.e., leaving the agglomerated mass so that it may be readily removed.

After the removing of the binder, the agglomerated masses are heated above the melting point to form a plurality of single, solid particles characterized as indicated hereinabove. One may, of course, heat to the melting point or above the melting point.

The second heating stage provides a plurality of distinct spherical solid metal particles predominately in the desired particle size.

After the heating stage the particles are cooled and the stop weld material removed. Frequently, it is only necessary to screen the particles in order to remove the stop weld material. If desired, one may wash or etch the materials.

The present invention will be more readily apparent from a consideration of the following illustrative examples.

Example I

In accordance with this example there was provided 2000 grams of wet copper powder having a melting of 1983° F. and having a particle size below 100 mesh. The copper powders were broken up into three groups, Group I having a density of 0.6 gram per cc., Group II having a density of 1.1 grams per cc. and Group III having a density of 2.0 grams per cc. The water content was adjusted as follows: Group I—water content of 25%, Group II—water content of 19.4% and Group III—water content of 15.0%.

To each mass was added 3% by weight of polyethylene glycol in the solid form. The materials were admixed by tumbling so that agglomerated masses or balls were formed having a size between $-14 + 20$ mesh. The time of admixing was as follows: Group I—admixed for 4 minutes, Group II—admixed for 10 minutes and Group III—admixed for 14 minutes. The out of size, agglomerated masses were removed and the remainder coated with 4% by weight of talc.

The coated masses were heated in dissociated ammonia at about 800° F. for 15 minutes, thereby volatilizing the polyethylene glycol and the water. The masses were then heated at 2100° F. for 15 minutes to form the uniform spherical solid particles of the present invention. The particle size was between $-20 + 30$ mesh, with about 85 to 95% of the particles in each group being within this range.

The particles were screened to remove the talc and washed in dilute ammonium hydroxide.

Example II

In a manner after Example I, 500 grams of aluminum powder was provided having a particle size below 100 mesh and having a melting point of 1220° F. To this was added 100 cc. of water and 6% by weight of a fluoride-containing aluminum brazing flux. The materials were admixed by tumbling for 8 minutes, thereby forming agglomerated masses. The masses were predominately in the particle size $-14 + 20$ mesh, with the out of size masses being removed by screening.

The masses were then coated with 3% by weight of talc and the coated masses heated in dissociated ammonia at 800° F. for 15 minutes to volatilize the brazing flux and the water. This was followed by heating at 1450° F. for 15 minutes to form the solid spherical particles of the present invention. About 85 to 95% of the spherical particles formed were in the size range $-20 + 30$ mesh. The particles were screened to remove the talc and washed in a weak hydrofluoric acid solution.

This invention may be embodied in other forms or carried out in other ways without departing from the spirit or essential characteristics thereof. The present embodiment is, therefore, to be considered as in all respects illustrative and not restrictive, the scope of the invention being indicated by the appended claims, and all changes which come within the meaning and range of equivalency are intended to be embraced therein.

What is claimed is:

1. A process for obtaining spherical metal powders of a predetermined size range which comprises:

(A) providing powdered metal particles having a particle size below 100 mesh,

(B) providing less than 10% by weight of a binder which volatilizes above 150° F. and at least 200° F. below the melting point of said metal particles,

(C) admixing said metal particles and binder by tumbling in the presence of from 5 to 35% by weight of moisture to form spherical agglomerated masses of metal particles and binder, and removing said spherical agglomerated masses having a desired size range,

(D) coating said removed masses with a weld preventing material,

(E) heating said coated masses in a protective atmosphere to a temperature above the volatilization temperature of said binder, and

(F) heating the resultant product in a protective atmosphere to form a plurality of substantially oxygen free, solid, spherical, coherent metal spheres having a predetermined size range.

2. A process according to claim 1 wherein said powdered metal particles are selected from the group consisting of copper and copper base alloys.

3. A process according to claim 1 wherein said pow-

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dered metal particles are selected from the group consisting of aluminum and aluminum base alloys.

4. A process according to claim 1 wherein said moisture is water.

5. A process according to claim 1 wherein said protective atmosphere is a reducing atmosphere.

6. A process according to claim 1 wherein said protective atmosphere is dissociated ammonia.

7. A process according to claim 1 wherein said step (E) completely volatilizes said binder.

8. A process according to claim 1 wherein the weld preventing material is removed after said step (F).

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