

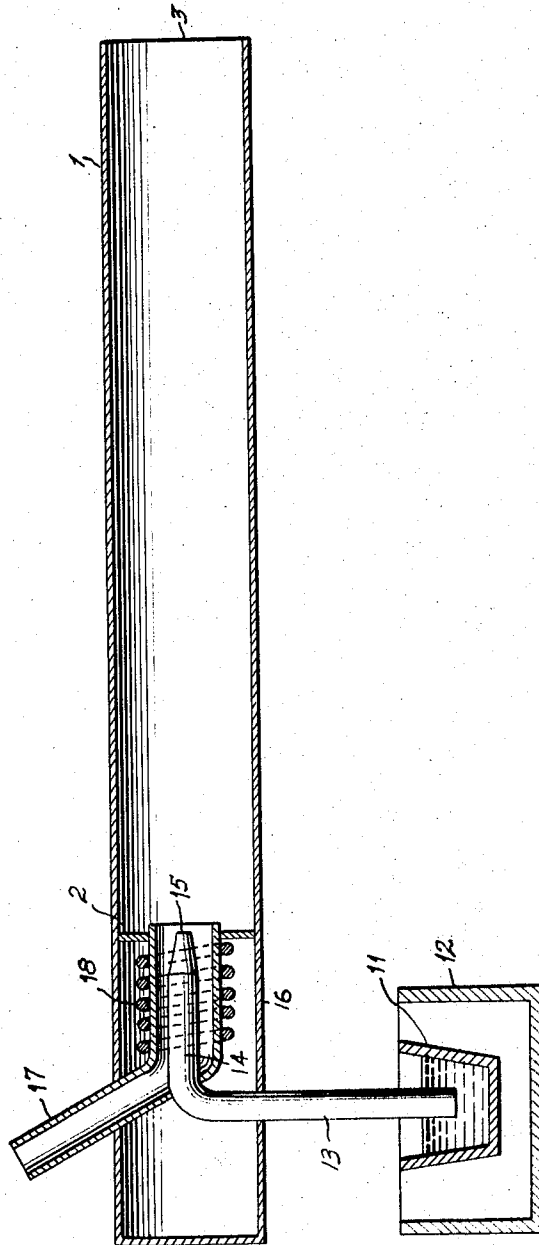
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PREPARATION OF SPHERICAL METAL POWDER

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PREPARATION OF SPHERICAL METAL POWDER

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This invention relates to the production of metal powders composed of spherically shaped particles. More particularly, the invention concerns a method for the manufacture of fine spherical aluminum powder.

Finely divided metal powders such as, for example, aluminum, aluminum alloy, copper, magnesium, and tin powders, have numerous industrial applications, including powder metallurgy, pyrotechnics, flares, and solid fuel components. In these and other applications, it is generally desired to obtain products which are free-flowing, possess a high packing density and smooth surface, and which are spherical in shape.

One of the known methods for the production of finely divided spherical metal powders is that of atomizing the molten metal, by means of a gas inert to the metal, into a closed chamber. In metal atomization as thus carried out, considerable stress has been laid on the necessity of avoiding surface oxidation of the molten metal, the thought being that the usefulness of the finished powder was impaired for some applications if the powder particles became excessively oxidized. It was also thought that the production of spherical metal powders, by the atomization of molten metal with inert gases containing small amounts of oxygen or nitrogen, was impeded by formation of metal oxides or nitrides through chemical reaction, causing clogging of the atomizing nozzle and other difficulties. Accordingly, elaborate purification systems have been proposed to remove impurities such as oxygen and nitrogen, especially from helium or argon inert gas, the purified inert gas then being recirculated to the main circulatory system.

In accordance with the present invention, however, it has been found that the presence of small amounts of oxygen during formation of particles from molten metal is not detrimental, provided oxidation of the metal is carefully controlled. The use of an atmosphere containing sufficient available oxygen to achieve some oxidation of the particles can even be advantageous in producing fine powder, since the resulting oxide film affords protection from ignition or detonation of metals which are otherwise difficult to handle with safety. The amount of available oxygen must be controlled very carefully, however, to avoid interference with normal surface tension forces in the particles of molten metal. A practical upper limit appears to be from about 0.2% to about 0.3% by volume of available oxygen.

When molten metal is atomized, the particle initially formed is typically elongated in shape owing to the action of the high velocity gas stream which tears it away from the stream of molten metal issuing from the atomizing nozzle. If the oxygen or moisture is present in sufficient amount, an oxide envelope immediately forms, preventing normal surface tension forces within the molten metal particle from pulling the particle into a spherical shape, the shape having the minimum surface area. If the metal does not form an oxide film, the initially formed metal particle will draw itself into a sphere, providing it remains substantially molten and free of external forces for a period of time sufficient to permit sphere formation. Another important consideration is that certain metals from which it is desired to produce

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spherical powder, notably aluminum and magnesium, are ordinarily pyrophoric in their unoxidized condition. Accordingly, at the earliest practical stage of the production of powder from such metals it is desirable to at least partially oxidize or otherwise protect the particle surfaces, in the interests of safety and for convenience in subsequent handling operations.

The present invention provides a novel technique for the production of metal powder, utilizing conditions which promote the formation of spherical particles (by avoiding interference with the natural forces of surface tension during the critical "spherodizing" operation), and producing a product which may be safely handled in air.

In accordance with this invention, fine spherical particles are produced by disintegrating molten metal into fine particles and keeping the particles substantially molten long enough for the effects of surface tension to cause formation of spherical particles, progressively oxidizing the particles to form a protective coating. This may be accomplished, for example, by atomizing molten metal with a high velocity stream of gas inert to the metal, spherodizing and cooling the metal particles in an atmosphere having a maximum available oxygen content insufficient to interfere significantly with the forces of surface tension, and continuing the oxidation of the particles in an atmosphere having an amount of free oxygen below that which will support ignition.

As employed herein, the term "available oxygen" means oxygen made available from gaseous oxygen compounds which may be present, through decomposition of such compounds by the molten metal, as well as oxygen which is present in the free, uncombined state.

Inert gas containing limited amounts of available oxygen, suitable for use in the spherodizing stage, is preferably produced by an exothermic reaction, and is customarily known as an exothermic gas. As produced, for example, by the controlled combustion of natural gas in a gas generator, a typical exothermic gas composition is:

CO ₂	-----	11-12% by volume.
H ₂ O	-----	Saturated at discharge temperature.
Combustibles		
(CO+H ₂ +CH ₄)	--	0.5% max.
O ₂	-----	No more than 0.3% (typically 0.2%).
N ₂	-----	Balance.

The desired sequence of steps may be carried out, in accordance with the invention, by discharging atomized particles of molten metal through a tubular shroud which provides a spherodizing zone, open to the air at the end of the tube opposite to that where the atomized particles are introduced. The shroud may be an elongated tube through which the particles are projected in a continuous stream from the feed end of the tube to the discharge end.

A gas such as the foregoing composition may be used for the atomizing gas, as well as for the atmosphere of the spherodizing zone; or the atomizing medium may be an inert gas which is substantially free of oxygen and water vapor, with the gas containing about 0.2-0.3% oxygen being admitted to the spherodizing zone through an independent inlet.

The atomizing gas employed in accordance with one aspect of the invention is accordingly an inert gas containing a controlled amount of available oxygen, hereinafter referred to as "substantially inert gas." This gas is discharged into the elongated zone, along with the disintegrated metal, providing within said zone an atmosphere containing available oxygen in an amount insufficient to interfere with normal surface tension forces of the particles causing sphere formation, and limiting

the rate of oxide formation during the passage of the particles through the zone in which sphere formation takes place. Thereafter, the limited amount of available oxygen in the gas serves as a means of providing controlled and progressive oxidation of the surfaces of the particles as they cool and are swept through the tube toward its exit.

The method of the invention is adapted for the production of spherical metal particles generally, including such metals as copper, magnesium, and aluminum and aluminum alloys. For purposes of illustration of the novel principles of the invention, reference will be made to aluminum and aluminum base alloys, including alloys with copper, tin, and magnesium.

The invention permits the production of spherical aluminum and aluminum alloy powders in improved yield, with greater economy, and having desirable characteristics of spherical shape, smooth surface, high packing density, and free-flowing properties.

In the practice of the invention, a metal such as aluminum or an alloy of aluminum, is melted in a resistance furnace and atomized by means of gas which is substantially inert to the metal, such as the aforementioned exothermic gas. An elongated metal tube may be employed, the diameter of which is small compared with its length, to provide a particle formation and oxidation zone. In this elongated zone, spherical particle formation takes place as the metal particles leave the atomization nozzle at high velocity, carried by a stream of the gas used for the atomization. As the stream of particles moves rapidly down the length of the tube, solidification and cooling take place, together with progressive oxidation of the particle surfaces as the particles approach the discharge end of the tube. Finally the spherical particles are withdrawn at the open end of the tube, and collected in a suitable collection system.

Thus, the portion of the tube nearest the atomizing nozzle provides a region wherein the spheres are allowed to freeze so as to fix their geometry and also to withdraw most of their heat energy as they pass through it.

The portion of the tube nearest the exit constitutes an oxidizing and cooling region. The metal spheres are swept along the length of the elongated tube by the action of the atomizing gas stream, passing through these two successive regions or sub-zones.

The method of the invention may be more readily understood by reference to the accompanying drawing, which is a lengthwise view of the apparatus in cross-section, showing the metal supply, atomizing system, and tube arrangement.

The apparatus as shown in the drawing comprises an elongated metal tube 1, providing a particle formation and oxidation zone. Tube 1 has a feed end, defined by a partition 2, a continuous interior bore and an exit end 3. The tube is long in comparison with its diameter, the relative dimensions being exemplified, for instance, by a tube 10" in diameter and 8 feet in length. Metal is heated in a pot 11 of a resistance furnace 12, the molten metal being withdrawn through pipe 13 into atomizing nozzle 14, which may be lined with a thin ceramic layer and which is provided with a ceramic tip 15, from which the molten metal is fed into tube 1. Surrounding the atomizing nozzle is a jacket 16 through which the atomizing gas is fed under pressure, causing disintegration of the molten metal. The gas is supplied through conduit 17, and the materials and nozzle may be heated by means of electrical heating elements shown generally at 18. The atomizing assembly projects through partition 2, which acts to seal off the particle formation zone of the tube. The finished metal powder is blown from the open end 3 of tube 1 and collected by any suitable collection system, not shown.

Specific operating conditions will vary with such factors as the particular metal being atomized, the orifice size of the nozzle, and temperature and pressure of the atomizing

gas. For purposes of illustration, however, using aluminum as an example, the metal temperature will be about 300° to 700° F. above its melting point; and the nozzle temperature is maintained at about 1300-1700° F.

The gas temperature in the spherodizing zone should be not less than about 600° F., and preferably in the range of 600-1200° F.

The following examples are illustrative of suitable operating conditions in the atomizing of commercial purity aluminum:

Example 1

Molten aluminum at about 1840° F. was admitted to the atomizing nozzle and disintegrated by a stream of exothermic gas having the composition previously referred to. A suitable spherodizing zone was created by ejecting the atomizing gas and entrained particles through an elongated tube (as shown in the drawing). The gas was supplied under pressure of 90 p.s.i., at a temperature of about 100° F. Nozzle temperature was approximately 1620° F.

The proportion of spherical particles was estimated to be about 60%, based upon microscopic examination.

Example 2

In the manner of Example 1, molten aluminum at a temperature of about 1520° F. was atomized with dry exothermic gas at 60 p.s.i., the nozzle temperature being about 1700° F. Comparable results were obtained.

Example 3

Following the procedure of Example 1, except that a manifold was added to the tubular shroud adjacent the nozzle and additional exothermic gas was thereby admitted to the spherodizing zone, aluminum at a temperature of about 1600° F. was atomized with gas at 600° F., 90 p.s.i. The use of this manifold arrangement was found to avoid pockets of reduced pressure and consequent turbulence adjacent the nozzle. The nozzle temperature was about 1500° F. and the temperature in the tube, 6 feet from the nozzle, was found to be 600° F.

Example 4

Using the manifold arrangement and the same atomizing gas temperature and pressure of Example 3, aluminum at a temperature of about 1820° F. was atomized to yield 50% spherical particles. The nozzle temperature was about 1350° F. and the oxygen content in the tubular shroud, 6 feet from the nozzle, was found to be about 0.2%.

The apparatus embodiment in the drawing shows the tube as cylindrical in configuration, but the tube can be flared outwardly toward the open end if desired. The length of the tube depends to some extent upon the pressure of the atomizing gas; and the requisite function it provides is to cause a shrouding of the metal particles with gas having a limited oxygen content. The tube cannot be too narrow, however, or the metal particles will coat the interior of the tube.

While present preferred embodiments of the invention have been illustrated and described, it will be appreciated that the invention may be otherwise variously embodied and practiced within the scope of the following claims.

What is claimed is:

1. Method of producing spherical metal powder, which comprises subjecting molten metal to the action of a high velocity stream of exothermic gas containing nitrogen and carbon dioxide, to disintegrate the metal into fine particles, said gas providing available oxygen in an amount insufficient to interfere with surface tension forces of the particles effecting sphere formation, and passing said gas stream and the particles of molten metal through a spherodizing zone to cause sphere formation and solidification of the spherical particles.

2. Method of producing spherical metal powder, which comprises dispersing fine particles of molten metal in a stream of exothermic gas consisting essentially of a mix-

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ture of nitrogen and carbon dioxide containing a small amount of oxygen not exceeding about 0.3% by volume, said gas providing available oxygen in an amount insufficient to interfere with surface tension forces of the particles effecting sphere formation, passing said particles and gas stream through a tubular spherodizing zone open to air at the end opposite the feeding end, causing solidification of spherical particles in said zone and progressive oxidation of the spherical particle surfaces, and collecting the finished particles at the open end of the zone.

3. The method of claim 2, in which the metal is a member selected from the group consisting of aluminum and aluminum alloys.

4. In the atomizing of molten aluminum to produce aluminum powder, the method which comprises discharging particles of molten aluminum from an atomizing nozzle, protecting the particles from premature oxidation while promoting the formation of spheres by maintaining said particles substantially in their molten condition within an atmosphere of exothermic gas consisting essentially of a mixture of nitrogen and carbon dioxide containing not more than about 0.3% oxygen by volume, said gas providing available oxygen in an amount insufficient to interfere with surface tension forces of the particles effecting

sphere formation, and cooling the spherodized particles in said atmosphere to cause solidification of the aluminum and progressive oxidation of the spherical particle surfaces.

5. The method of claim 4, in which said exothermic gas contains about 11-12% carbon dioxide, about 0.2% oxygen, balance substantially nitrogen.

6. The method of claim 4, in which said exothermic gas contains not more than about 0.5% combustibles.

7. The method of claim 1, in which said exothermic gas contains about 11-12% carbon dioxide, balance substantially nitrogen.

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