

Dec. 20, 1966

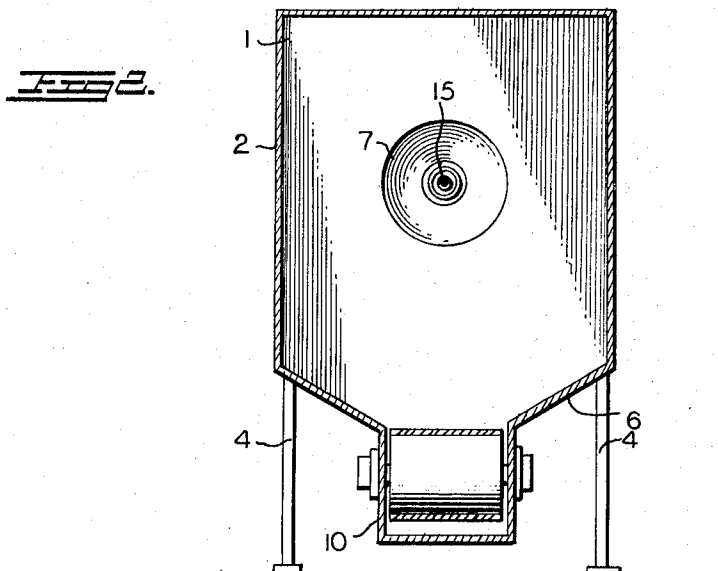
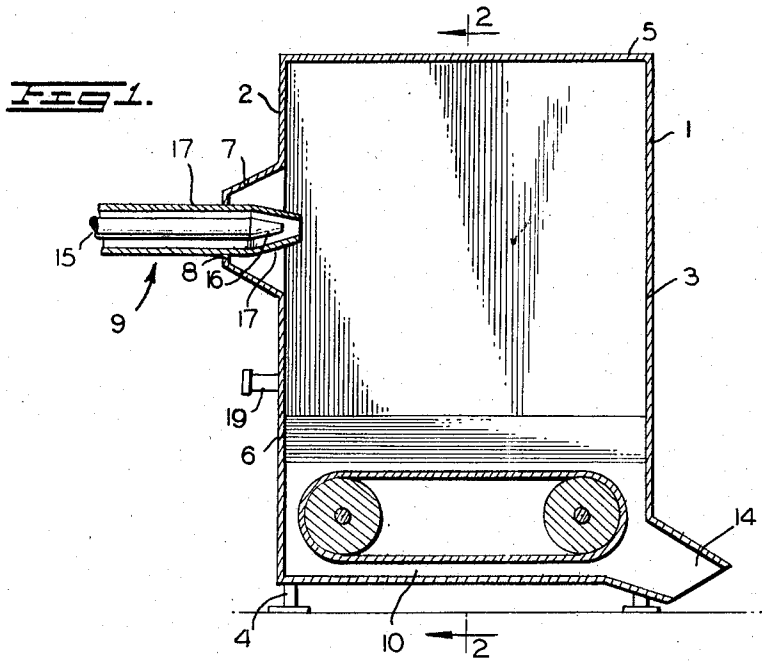
W. P. WOOSLEY ETAL

3,293,333

PRODUCTION OF FINE SPHERICAL METAL PARTICLES

Filed Aug. 16, 1962

3 Sheets-Sheet 1



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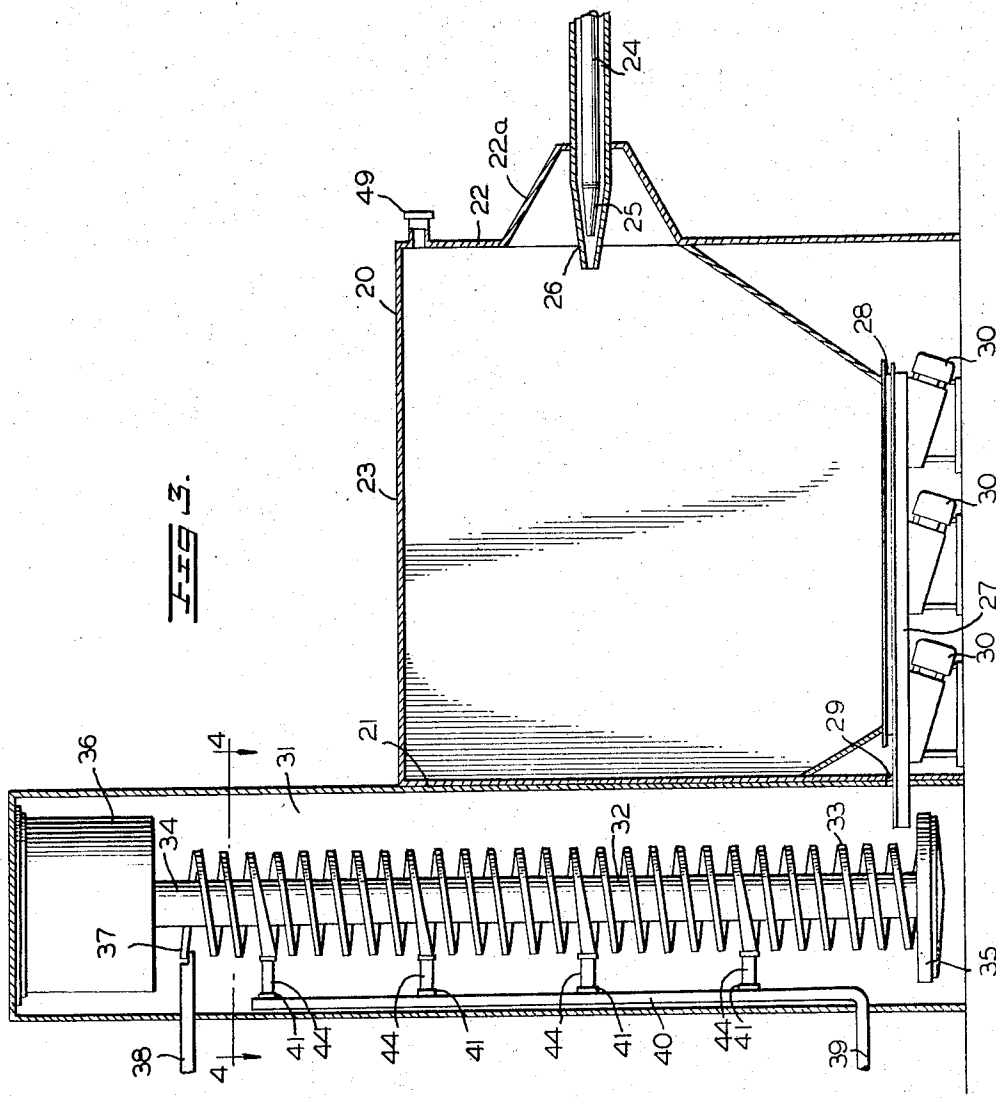
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3 Sheets-Sheet 2



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FIG 4.

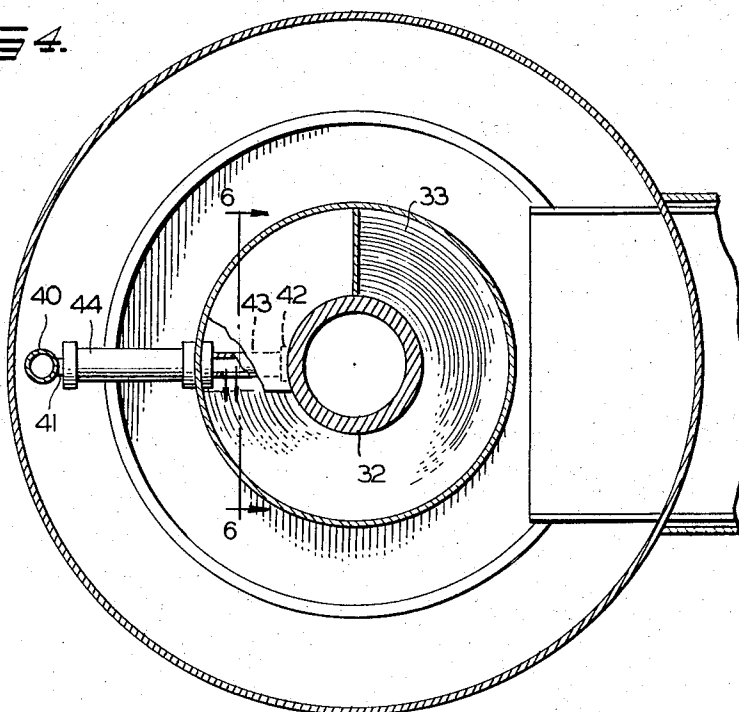


FIG 5.

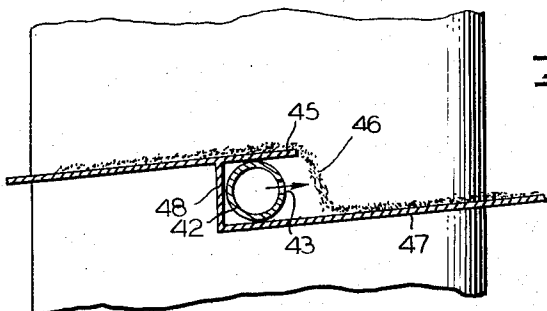
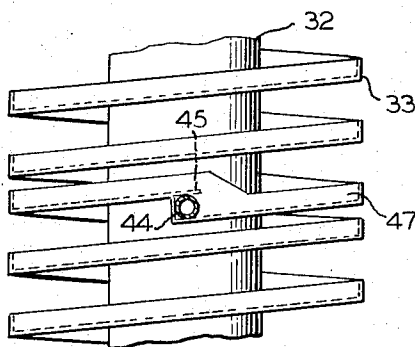


FIG 6.

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PRODUCTION OF FINE SPHERICAL METAL PARTICLES

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Filed Aug. 16, 1962, Ser. No. 217,414
11 Claims. (Cl. 264—12)

This invention relates to the production of metal powders composed of particles spherical in shape. More particularly, the invention concerns a method for the continuous manufacture of fine spherical aluminum particles by atomization of the molten metal with an inert gas and subsequent exposure of the atomized metal to an inert gas providing controlled oxidation of the metal surface. The invention is especially concerned with spherical aluminum powders.

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

One of the known methods of producing finely divided 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 carried out in this fashion, the art has laid considerable stress on the necessity of avoiding surface oxidation of the molten metal, the thought being that the usefulness of the finished powder was greatly impaired when the powder particles became coated with oxide. It was also believed by those skilled in the art that the production of spherical metal powders from molten metal by atomization with inert gases which might contain small amounts of oxygen and nitrogen was impeded by formation of metal oxides and nitrides by chemical reaction, causing clogging of the atomizing nozzle and other difficulties. Accordingly, elaborate purification systems were proposed to remove impurities such as oxygen and nitrogen, especially from argon or helium inert gas media, the purified inert gas then being recirculated to the main circulatory system.

In accordance with the present invention, it was found, surprisingly and unexpectedly, that the presence of small amounts of oxygen during formation of particles from the molten metal is not detrimental, provided that the degree and/or rate of oxide formation on the surface of the molten metal is controlled. Control of the degree and/or rate of oxide formation, according to the invention, is accomplished by atomizing the metal into an inert gaseous atmosphere in which solid particle formation takes place, said atmosphere containing a small amount of available oxygen, sufficient to achieve a degree of surface oxidation which will protect the metal from ignition or detonation, but insufficient to interfere with the normal surface tension forces tending to draw the molten metal particle into spherical shape.

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

When molten metal is atomized, the particle initially formed is 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 oxygen or moisture is present in sufficient amount, an oxide envelope immediately forms, preventing the normal

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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 molten and not subject to external forces greater in magnitude than its own surface tension forces for a period of time sufficient to permit sphere formation.

Atomization breaks the metal into small particles and maintains it in molten condition long enough to form spheres. Small particle size is a factor favoring sphere formation.

The method of the invention is adapted for the production of spherical metal powders generally, including such metals as copper, magnesium and aluminum and their alloys. For purpose of illustration of the novel principles of the invention, aluminum and aluminum base alloys, including alloys of aluminum with copper, tin, and magnesium, will be referred to herein.

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 accordance with one aspect of the invention, there is provided a method for the formation of fine spherical metal particles which comprises forming a body of molten metal, subjecting said body of molten metal to the action of a high velocity stream of a gas inert to said metal to disintegrate the metal into fine particles, and discharging said molten particles and said inert gas stream into an enclosed cooling zone comprising an atmosphere of a gas inert to the metal and containing available oxygen in an amount insufficient to interfere with normal surface tension forces within the molten metal particles causing sphere formation.

The atomizing gas may itself be either a gas inert to the metal and containing no oxygen, or it may be a gas inert to the metal and containing a limited amount of available oxygen, identical with or similar in composition to the atmosphere of the cooling zone.

The inert gas containing limited amounts of available oxygen 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% maximum.
O ₂ -----	Less than about 0.3%.
N ₂ -----	Balance.

In accordance with another aspect of the invention, there is provided a multistage method, which is adapted for continuous operation, for the production of spherical metal powders. In this method, the formation of spherical particles from molten metal is divided into two stages, with controlled access of oxygen to the metal in each stage, thus providing for progressive oxidation.

In the first stage, a body of molten metal is subjected as before to the action of a high velocity stream of a gas inert to the metal to disintegrate the metal into fine particles, the molten particles and said gas stream being discharged into a first zone comprising an atmosphere of the inert gas. The inert gas contains available oxygen in an amount insufficient to interfere with normal internal surface tension forces in the particles causing sphere formation. The spherical particles are partly cooled, collected and transported to a second zone in which further oxidiz-

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ing and cooling takes place in the presence of an atmosphere with a controlled amount of free or available oxygen, the amount of the oxygen being below that which will support ignition. The amount of oxygen that may cause difficulty in this regard may be between about 5% and about 10%, but this will depend upon particle size and other factors.

The single stage method may be performed as a batch operation, utilizing an apparatus providing an enclosed cooling zone in the form of a closed chamber into which the gas and molten particles are discharged, the velocity of the molten particles being rapidly diminished upon entry in the chamber. The chamber is filled with the inert gas containing a limited amount of available oxygen. Cooling and sphere formation take place, the metal particles collecting at the bottom of the chamber on a suitable conveyor. The initial atmosphere in the chamber may be replaced by an atmosphere of an inert gas containing a controlled amount, e.g. about 5%, of free oxygen, to complete oxidation of the particle surfaces in the same chamber. The conveyor transports the finished particles out of the chamber to a suitable collecting system.

The preferred embodiment of the invention comprises a two-stage method and apparatus, whereby the metal is atomized into a first or cooling zone formed by a chamber provided at the bottom thereof with a horizontal type vibratory conveyor, upon which the spherical metal particles collect and are transported to a second chamber, which serves a further cooling and oxidizing zone. Both the first and second zones are thus separated and each is provided with means for supplying inert gas containing available oxygen. A slight pressure is maintained inside to prevent leakage of outside air.

The two-stage method is especially adapted for continuous operation, the metal particles moving progressively through the first cooling and sphere formation zone containing an inert gas having a limited amount of oxygen, as previously described, thence to a second cooling and oxidizing zone containing an inert gas having about 5% oxygen, and thence to a collecting system where the particles become exposed to the outside air.

In the second zone, the metal particles, while mechanically agitated, are moved continuously through the zone, and are subjected at intervals to a mechanical cascading or tumbling step, in order to expose the particle surface fully and effectively to the action of the oxygen containing gas in the second zone. During these intervals the cascading particles are subjected to the action of streams of the oxygen containing gas. This cascading action is preferably accomplished, in accordance with the invention, by means of a spiral conveyor, described more fully below. The particles passing upwardly in this conveyor, which is of the vibratory type, periodically traverse portions of the spiral at which there is provided a steplike interruption in the helical conveyor surface. Thus, the particles are at intervals subjected to a downward movement during which they form a cascade, and the cascading particles are subjected to the action of a stream of inert gas containing available oxygen, said gas stream being applied in a direction transverse to the direction of the falling particles.

Accordingly, the objects of the invention include the provision of a method for the production of spherical metal powders by atomization and subsequent controlled oxidation of the metal particle surface by intermittent exposure of a cascade of the particles to the action of an inert gas containing a small amount of available oxygen.

These and other objects will be apparent from the detailed description below, reference being made to the accompanying drawings, in which:

FIG. 1 is a side elevation, in cross-section, of a single chamber apparatus suitable for batch operation;

FIG. 2 is a vertical sectional view, taken along the line 2—2 of FIG. 1;

FIG. 3 is a side elevation, partly in cross-section, show-

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ing a multi-chamber apparatus, suitable for continuous operation;

FIG. 4 is an enlarged cross-sectional view along the line 4—4 of FIG. 3, showing the detail of the gas treatment means;

FIG. 5 is an enlarged fragmentary detailed sectional view of a portion of the helical conveyor, showing the step construction; and

FIG. 6 is an enlarged detailed sectional view taken along the line 6—6 of FIG. 4.

As shown in FIG. 1, the single chamber apparatus includes a chamber 1, made of any suitable material, such as steel sheet, and of any desired shape, preferably rectangular shape. Chamber 1 is provided with side walls 2 and 3, a closed top 5, and a hopper type bottom shown generally at 6. The chamber is supported on legs 4. In sidewall 2 there is located in the center portion thereof a flared outwardly projecting portion 7, having a terminal member 8 serving as a means of mounting the atomizing assembly 9. The lower portion of the chamber 1 terminates in a recessed channel section 10, in which there is suitably mounted a conveyor, which may be of any suitable type, such as a belt conveyor, as shown, or a vibrating type conveyor. Channel section is provided, at the end opposite to the side wall containing the atomizing assembly, with a discharge chute 14, by means of which metal powder collecting on the conveyor is discharged to the atmosphere. Driving means (not shown) for the conveyor cause movement of the upper portion of the conveyor belt towards the discharge chute. The conveyor is not in operation during a run. The atomizing assembly includes an atomizing nozzle 15 which may be a metal pipe lined with a thin ceramic layer, and which is provided with a ceramic tip 16 from which the molten metal from a source not shown is fed into chamber 1. Surrounding the atomizing nozzle 15 is a jacket 17 whereby inert gas, which is preferably preheated, is fed into the atomizing assembly under pressure, causing disintegration of the molten metal. Inlet 19 provides for introduction of replacement gas.

In the operation of the embodiment shown in FIGS. 1 and 2, molten metal, such as molten aluminum, is fed into atomizing nozzle 15, while a stream of an inert gas containing a limited amount of available oxygen, such as an exothermic gas previously described, is fed into the surrounding jacket under pressure sufficient to cause disintegration of the molten metal stream into small particles, which discharge into the interior of chamber 1. The metal particles, under the action of surface tension forces, draw into spherical shape, fall and collect on the upper surface of the conveyor belt located at the bottom of the chamber. When a run is completed, the conveyor is operated causing the spherical metal powder to be discharged through chute 14 into a suitable receiver. The batch operation may be carried out using either an inert gas containing no oxygen, or a gas containing a limited amount, for example 0.2% by volume, of oxygen, as the atomizing gas. The chamber may be initially filled with an inert gas containing a limited amount of available oxygen of the same type as used for atomization, such as an exothermic gas. In order to complete the oxidation of the metal particles in the same chamber, the gas initially present, and the gas introduced into the chamber via the atomizing nozzle, may be replaced by blowing into the chamber a fresh supply of an inert gas containing a slightly larger amount of available oxygen, such as, for example, about 5% by volume, and the oxidation completed in the chamber by exposure of the metal particles to the cooling and oxidizing action of the replacement gas. When the run is completed, the conveyor is operated to discharge the particles to the outside.

The preferred embodiment of the invention is the multi-chamber system shown in FIGS. 3 and 6 inclusive.

Referring to FIG. 3 the apparatus comprises an atomizing chamber 20 provided with walls 21 and 22 and closed

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top 23. In the central portion of wall 22 there is mounted the atomizing assembly, on an outwardly flared projecting wall portion 22a, including an atomizing nozzle 24 which may be a metal pipe lined with a thin ceramic layer, and which is provided with a ceramic tip 25 from which molten metal is fed, from a source not shown, into chamber 20. Surrounding the atomizing nozzle 24 is a jacket 26 whereby inert gas, which is preferably preheated, is fed into the atomizing assembly under pressure, causing disintegration of the molten metal. Inlet 49 provides for introducing additional gas.

Located in the bottom portion of chamber 20 is a vibrating conveyer 27, comprising a trough 28 having a generally flat central configuration, upon which the falling metal particles collect. The trough may be either horizontal or inclined at a slight angle toward the discharge opening 29 located at the lower end of wall 21 of the chamber. The conveyer 27 is actuated by a series of electromagnetic vibrators 30 which are of conventional type including an electromagnet and armature. The action of the conveyer depends upon the principle that under controlled vibration a loose bulk material will travel over a surface by itself, with its speed and degree of vibration unit. The action is that of pulling the trough downward and backward, leaving the load momentarily suspended in space. It then falls vertically, landing slightly, up to about $\frac{1}{8}$ " ahead of its earlier position, producing an effect of almost continuous flow, which can be increased by sloping the trough a few degrees toward the discharge. The conveyer projects a short distance and discharges into adjacent chamber 31 through communicating discharge opening 29.

Chamber 31 houses a vertical helical vibratory conveyer 32, which may be of any suitable type, such as disclosed, for example in U.S. Patent 2,658,286. In the arrangement shown in FIG. 3, the helical or spiral conveyer consists of a spirally arranged conveyer trough 33 mounted on a central tube 34 and extending from the lower end to the upper end of tube 34. The lowermost trough portion is provided with an annular trough 35 for receiving bulk feed material. Located to the upper portion of the conveyer, is an actuating means shown generally at 36, which includes a pair of symmetrically mounted electromagnetic reciprocating motors energized by current impulses, which impart their magnetic forces to the conveyer to reciprocate it in such manner that the vibrations cause the material to flow in a circular path about the axis of the central tube 34 and up the inclined path provided by the helical or spiral trough. The top of the uppermost conveyer flight is provided with a laterally extending or tangential discharge trough 37 which discharges the material into an exit trough or tube 38, whereby it is transported to collecting means not shown, located outside chamber 31.

Chamber 31 functions as a further cooling and oxidizing chamber, and is accordingly provided with means for introducing a supply of suitable treating gas, such as, for example, an inert gas containing a limited amount of available oxygen. The means for introducing gas comprises an inlet 39, connecting with a vertically extending central supply pipe or header 40, located inside chamber 31, and running parallel to the central conveyer tube 32. Located at suitable intervals is a series of nipples 41, positioned opposite lengths of pipe 42, fastened to central tube 32 and extending laterally therefrom, said pipe lengths being of approximately the width of the conveyer trough 33. Each pipe length contains a horizontal slot 43 through which the gas issues under slight pressure. The slotted pipes are connected to the nipples by flexible connectors 44 so that the vibratory action of the conveyer will not affect the gas supply system, by causing loosening of connections, leakage, and the like.

In accordance with a novel feature of the invention, there are provided at intervals in the conveyer trough 33, discontinuities 45, in and extending across the upper sur-

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face thereof over which the ascending material falls in the form of a cascade 46, as shown in FIG. 6, the distance of fall depending upon the depth of the step formed by the discontinuities. The profile of the discontinuity in the helical trough is shown in FIGS. 5 and 6. The discontinuity 45 is formed by a portion of the spiral trough which overhangs the continuing portion 47 of the trough, upon which the cascading material falls to resume its upward journey. The height of the cascade is slightly greater than the outside diameter of the slotted pipe 42. The latter is positioned in a recess slightly greater than the outside diameter of the slotted pipe 42, and formed by wall 48 which extends from the edge of trough position 47 to the under surface of the preceding portion of the trough. The slotted pipes 42 extend across the under surface of the trough positioned just behind and in a direction parallel to the edge of the discontinuity 45, with the slots 43 positioned so that the gas issuing therefrom will traverse the cascade of falling particles 46, thus cooling, oxidizing and further agitating of said particles.

In the embodiment shown in FIG. 3, there are four cascades and corresponding sets of slotted gas treatment pipes.

It will be understood that the principle upon which the embodiment shown is based may be similarly applied in other types of spiral conveyers, including those operating with a downward flow of material under gravitational forces. Thus, a conventional gravity spiral conveyer can be adapted for the purpose of the invention by providing discontinuities at intervals in the trough thereof, in conjunction with a slotted pipe gas treatment system of the character described.

In the continuous operation of the embodiment shown in FIGS. 3-6 molten metal, such as aluminum, is supplied to the atomizing nozzle and inert gas containing either no oxygen or a limited amount of available oxygen, such as exothermic gas, is supplied to the surrounding jacket, serving to disintegrate the stream of molten metal into small particles which draw into spherical shape in the first or cooling chamber 20. The particles collect on the conveyer by means of which they are continuously moved to chamber 31. Treating gas comprising an inert gas containing about 5% oxygen by volume is introduced into chamber 31 via the slotted pipe system, or additionally by direct introduction into the chamber, under slight pressure to prevent leakage of outside air. The gas envelops the material moving upward on the spiral conveyer, and also contacts the material descending in the respective cascades formed by the discontinuities in the trough surface. The cooled and oxidized metal particles are then discharged into suitable collecting devices and stored in any desired manner.

The choice of operating conditions will depend upon the metal being atomized, size of nozzle orifice, gas temperatures and pressures, and other factors. For purposes of illustration, but not of limitation, in the case of aluminum, nozzle temperatures may range from about 1300° to 1700° F. Gas temperatures in the first chamber may range between about 600° and about 1200° F., but these figures are indicative only, and are subject to wide variation. In the second chamber, the gas temperature may be in the range 600° to 900° F., again with wide variations.

What is claimed is:

1. Method for the production of fine spherical metal particles which comprises subjecting molten metal to the action of a high velocity stream of gas inert to said metal, to disintegrate the metal into fine particles, discharging the molten metal particles and said gas stream into an enclosed cooling zone having an atmosphere of exothermic gas containing nitrogen and carbon dioxide, said exothermic gas providing available oxygen in an amount insufficient to interfere with normal surface tension forces of the particles causing sphere formation, allowing the particles to assume spherical shape in said atmosphere, and exposing the spherical particles to inert gas containing about 5% free oxygen by volume.

2. Method for the production of fine spherical metal particles which comprises subjecting molten metal to the action of a high velocity stream of gas to disintegrate the metal into fine particles, discharging the molten metal particles and said gas stream into an enclosed cooling zone, the velocity of the particles being rapidly reduced upon entry into said zone, both the atmosphere in said zone and the gas employed for disintegration consisting essentially of exothermic gas containing nitrogen and carbon dioxide, allowing the particles to assume spherical shape in said atmosphere, exposing the spherical particles to inert gas containing about 5% free oxygen by volume, and removing the particles into air.

3. The method of claim 1 in which the metal is selected from the group consisting of aluminum and aluminum alloys.

4. The method of claim 1 in which the gas employed for disintegrating the molten metal is an exothermic gas, consisting essentially of a mixture of nitrogen and carbon dioxide containing less than about 0.3% oxygen by volume.

5. Method for the production of fine spherical metal particles which comprises subjecting molten metal to the action of a high velocity stream of gas inert to said metal, to disintegrate the metal into fine particles, discharging the molten metal particles and said gas stream into an enclosed cooling zone, having an atmosphere of exothermic gas containing nitrogen and carbon dioxide, said exothermic gas providing available oxygen in an amount insufficient to interfere with normal surface tension forces of the particles causing sphere formation, the velocity of the particles being rapidly reduced upon entry into said zone, then replacing the atmosphere of said zone with inert gas containing about 5% free oxygen by volume to further cool and oxidize the spherical particles formed, and recovering the spherical metal particles.

6. Method for the production of fine spherical metal particles which comprises subjecting molten metal to the action of a high velocity stream of gas inert to said metal, to disintegrate the metal into fine particles, discharging the molten metal particles into a first enclosed cooling zone having an atmosphere of exothermic gas containing nitrogen and carbon dioxide, said exothermic gas providing available oxygen in an amount insufficient to interfere with normal surface tension forces of the particles causing sphere formation, the velocity of the particles being rapidly reduced upon entry into said first zone, allowing the particles to assume spherical shape and partly cool in said first zone, transporting the spherical particles to a second enclosed cooling and oxidizing zone, exposing the particles in said second zone to inert gas containing about 5% free oxygen by volume, and removing the particles from said second zone into air.

7. The method of claim 6, in which the gas used for disintegrating the metal and the atmosphere of said first zone both are exothermic gas consisting essentially of a mixture of nitrogen and carbon dioxide containing less than 0.5% combustibles by volume.

8. Method for the production of fine spherical metal particles which comprises disintegrating molten metal into fine particles, discharging the molten metal particles into a first enclosed cooling zone having an atmosphere of exothermic gas containing nitrogen and carbon dioxide, said exothermic gas providing available oxygen in an amount

insufficient to interfere with normal surface tension forces of the particles causing sphere formation, the velocity of the particles being rapidly reduced upon entry into said first zone, allowing the particles to assume spherical shape and partly cool in said first zone, transporting the spherical particles to a second enclosed cooling and oxidizing zone, moving said particles upwardly through said second zone in a stream, interrupting said stream at intervals to produce a downward cascading of the particles, subjecting the downwardly cascading particles to the action of inert gas containing about 5% free oxygen by volume, and recovering the metal particles.

9. Method for the production of fine spherical metal particles which comprises disintegrating molten metal into fine particles, discharging the molten metal particles into a first enclosed cooling zone having an atmosphere of exothermic gas containing about 11-12% carbon dioxide by volume, balance substantially nitrogen, said exothermic gas providing available oxygen in an amount insufficient to interfere with normal surface tension forces of the particles causing sphere formation, the velocity of the particles being rapidly reduced upon entry into said first zone, allowing the particles to assume spherical shape and partly cool in said first zone, transporting the spherical particles to a second enclosed cooling and oxidizing zone, moving said particles upwardly through said second zone in a spirally ascending stream, interrupting said stream at intervals to produce a short downward cascading of the particles, subjecting the downwardly cascading particles during their fall to the action of gas flowing in a direction transverse to the direction of fall of the cascade, the latter gas containing about 5% free oxygen by volume, balance substantially nitrogen and carbon dioxide, and recovering the metal particles.

10. The method of claim 9 in which the metal is selected from the group consisting of aluminum and aluminum alloys.

11. Method for the treatment of fine spherical metal particles which comprises moving a stream of said particles upwardly through an enclosed zone in a spirally ascending stream, interrupting said stream of particles at intervals to produce a short downward cascading of the particles, subjecting the downwardly cascading particles during their fall to the action of inert gas containing about 5% free oxygen by volume, the upward movement of the stream of particles being resumed after each interruption.

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