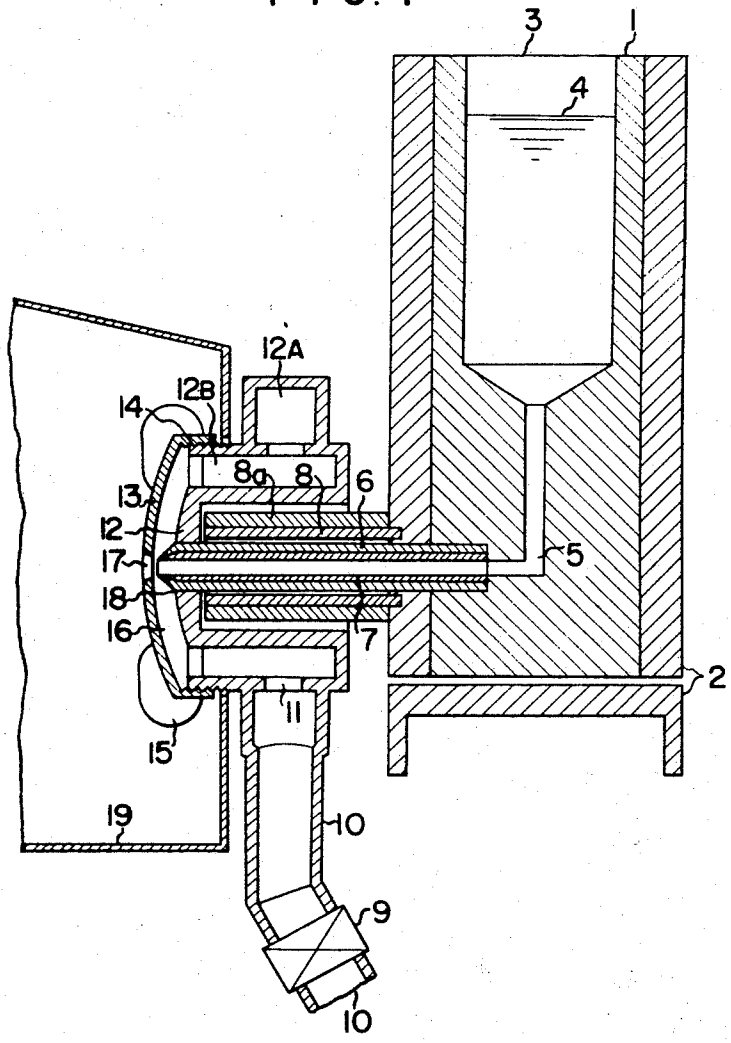
March 4, 1969 MICHINOSUKE AIKAWA ETAL 3,430,289

APPARATUS FOR PREPARING HIGH PURITY FINE POWDER OF LOW-MELTING METALS

Filed Nov. 1, 1965 Sheet 1 of 2

FIG. 1



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Filed Nov. 1, 1965 Sheet 2 of 2

FIG. 2(A)

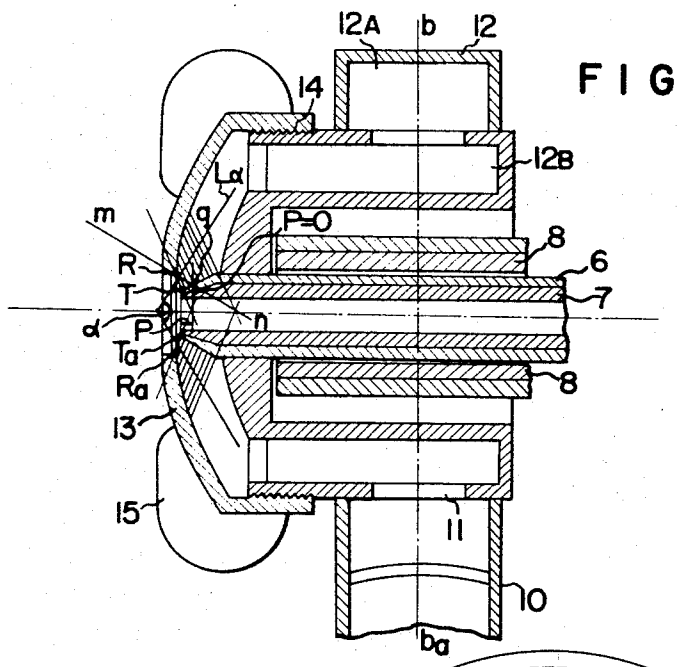
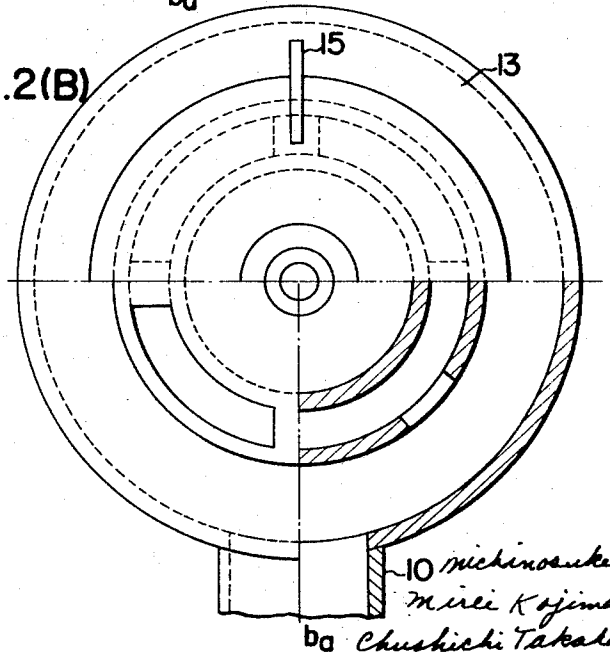


FIG. 2(B)



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APPARATUS FOR PREPARING HIGH PURITY FINE POWDER OF LOW-MELTING METALS

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ABSTRACT OF THE DISCLOSURE

An apparatus for preparing a high purity fine powder of a metal selected from the group consisting of zinc, lead, tin, cadmium, antimony, bismuth, aluminum and similar metals having a relatively low melting point. A crucible of refractory material has a uniform diameter nozzle connected to the lower end of the chamber therein below the level thereof so that molten metal is ejected through the nozzle by the pressure of the metal in the crucible. A nozzle box surrounds and supports the nozzle, and means are provided for supplying air under pressure of at most 1.5 kilograms per centimeter. The air supply means comprises an apertured cap adjustably mounted on the nozzle box in front of the nozzle with the cap and nozzle forming a ring-shaped cap air chamber inside of the cap. A ring-shaped inner air chamber is provided in the nozzle box which opens into the cap air chamber, and an outer ring-shaped air chamber around the nozzle box opens into the inner ring-shaped chamber. Air under pressure is supplied to the outer air chamber. A chamber is provided in front of the cap to recover the powdered metal.

The present invention relates to improved apparatus for preparing high purity fine powder of a metal having a relatively low melting point such as, for example, zinc, tin, lead, cadmium, antimony, bismuth, thallium, tellurium, selenium, aluminium or the like.

An essential object of the present invention is to provide an apparatus for preparing fine powders of high purity metals having homogeneous properties and containing only slight oxide such impurities such as oxide films, iron and the like, said oxide films being liable to occur on the surfaces of the metal powders, and said iron and the like being able to be alloyed with a metal to be treated such as zinc or aluminum, on an industrial scale with high efficiency and high production yield by melting a metal to be treated, for example, electrolytic metal which has been obtained by hydro-metallurgy as raw-material or a metal having a high purity corresponding to that of said electrolytic metal and having been obtained by another smelting process and atomizing said molten metal by utilizing compressed air of relatively low pressure as a medium of atomization.

It is true that production of various kinds of metal powder by the molten metal atomization method is widely-known and can never be recognized as a new technique. It can be widely applied to many kinds of metals including both simple metals and alloys as long as they are meltable, for example, to simple metals such as iron, copper and aluminum and to alloys such as brass, stainless steel and the like. Thus, it has been tested widely and powders of some metals are produced by this method on an industrial scale. Since the above mentioned metals and alloys are of low-melting metals, it is not difficult to produce their powders by said molten metal atomization method.

However, it must be noted that the properties and the quality or the cost of the produced metal powders are

generally different depending on the pulverizing method. For example, in the case of production of zinc powder, most of the metal powders such as zinc powders presently available on the market are generally prepared by the evaporation method which belongs to the methods of preparing powders from the vapour phase. The metal powder prepared by the evaporation method under ordinary conditions is always inferior to the zinc owder obtained by the atomization method. The former contains much more zinc oxide than the latter, and, in addition, as long as zinc powder is produced as a by-product of an evaporation process, or prepared from such waste raw materials as galvanizing dross, skimmings and the like, the purity of the resultant powder is inherently lower than that obtained by the atomization process. That is, the content of impurities such as cadmium, lead and the like is always higher than that obtained from the latter method. Moreover, it is evidently disadvantageous to produce zinc powder from the vapour phase according to the evaporation method by use of such high purity metal for raw material as electrolytic zinc in comparison with the atomization method, as regards cost, yield and the like. For this reason, the method is considered to have some aspects which are unsuitable for applying to the production of zinc powder used for precipitant (in hydrometallurgy), reducing agent (in dyestuffs industry) and for materials required in powder metallurgy, sherardizing, painting and the like.

Hithertofore, aluminum powder has been obtained in the state of granular particles or flake type of powder by stamp-mills method, ball-mills method or atomization method. When the powder obtained as mentioned above is used for spraying paint, it is usually worked so as to be flaky form in order to make said powder assume a metallic luster and a phenomenon of "leafing."

However, when said metal powder is to be used for powder metallurgy, a large amount of air is liable to mix into bodies of the mold according to the size and shape of the particles. Furthermore, pressing and sintering operations are disturbed by the oxide surface films and fatty acid (stearic acid) which is a lubricating agent used in the pulverization of the raw metal. Consequently, atomized products obtained by the atomization method, causing only slight oxidation, are appropriate.

Furthermore, tin powder has been prepared according to any one of various methods such as shotting, graining, atomization, chemical precipitation, or electrodeposition. In this case, the fine tin powder adapted for powder metallurgy work can usually be prepared according to said atomization, chemical precipitation, or electrodeposition methods. Among these methods, the atomization method is superior to the others, because it is possible to obtain fine tin powder containing only slight oxide on an industrial scale and with small cost more easily than according to said other methods.

Moreover, lead powder and cadmium powder have been usually prepared by shotting, graining, or the atomization method. However, the atomization method is the most appropriate to yield powders of sufficient fineness and high purity on an industrial scale and at small cost.

Antimony powder and bismuth powder have been usually prepared by pulverizing method, because said metals are very brittle. However, in order to obtain said powder on an industrial scale at small cost, the atomization method is superior to the pulverizing and electrodeposition methods.

Thallium powder, tellurium powder and selenium powder have been usually prepared by electrodeposition or a chemical precipitation method of hydro-metallurgy, but the atomizing method may be effectively utilized for preparing said metal powders with appropriate results.

It has long been required to perfect an economical and

mass-productive method for producing powders of sufficient fineness and high purity which have a homogeneous property and contain as small a quantity of oxide as possible, said method having additionally a capability of adjusting granularity distribution within any range.

This invention seeks to satisfy this requirement and makes it possible to prepare powders of various metals with very simple and economical apparatus utilizing a specially devised atomization method.

According to the present invention, briefly stated, there is provided an apparatus for carrying out a method for preparing fine powder of high purity metal by the atomization technique, which method comprises ejecting a molten metal to be powdered by means of air at a relatively low pressure of at the most 1.5 kilogrammes per square centimetre through a nozzle of relatively large bore diameter lined with a refractory material, the length of nozzle being selected from a wide range of values, and the ejection angle being an obtuse angle in the range of from 100 to 130 degrees.

The nature, principle, and details of the invention will be more clearly apparent from the following description with respect to preferred embodiments of the invention, when taken in conjunction with the accompanying drawings in which like parts are designated by like reference characters, and in which:

FIG. 1 is a side elevational view, in vertical section, showing the assembled state of an embodiment of the atomization apparatus according to the invention for atomizing molten metal;

FIG. 2 (A) is a side elevational view, in vertical section, showing an essential part of the atomization apparatus; and

FIG. 2 (B) is a front elevational view of the apparatus shown in FIG. 2 (A), in which view the lower right quadrant is a section taken along a plane indicated by the line b–b_a in FIG. 2 (A), and the lower left quadrant is a view with the cap removed.

Referring to FIG. 1, the apparatus shown therein is provided with a cylindrical crucible **1** made of carbon, which is provided about its outer cylindrical surface and bottom with heat preserving means **2**. The crucible **1** has in its interior a reservoir **4** into which molten metal can be poured through a pouring opening gate **3**, and from which molten metal is supplied through a tubular sprue runner **5** which runs vertically downward from the bottom of the reservoir **4** and bends at a right angle to become horizontal. This horizontal part of the sprue runner is connected to a nozzle **6** made of mild steel, the outer diameter of which gradually decreases toward its tip, and which is lined with a cylindrical inner tube **7** made of special refractory material. An outer tube **8** made of a special refractory material is fitted around the nozzle **6** and provided with a heating and heat preserving means **8***a*. Compressed air is led into a cap air chamber **16** by way of a blast pipe **10** and at least one inlet port **11**.

Compressed air is supplied from a compressor not shown through a pressure regulator **9**, a blast pipe **10**, an outer air chamber **12A** and an inner air chamber **12B** which are formed around a nozzle box **12** into a cap air chamber **16** which is formed between said nozzle box and a cap **13** fitted at its threaded portion **14** onto said nozzle box to cover the front of said nozzle box, said threaded portion being used for controlling atomization and detachable and closely controllable in fine forward and rearward adjustment. Reference numeral **15** designates an adjusting handle. Thus, by turning the cap **13**, the space between the nozzle **6** and the air ejection opening is controllable.

The tip of the nozzle **6** is fitted in the central bore **18** in the center of the nozzle box **12** so that the gradually decreasing diameter part of said tip protrudes into the interior of the cap air chamber **16** and then is fixed in tight contact with the nozzle box to form the compressed air nozzle for atomization. A dust chamber **19** is provided for recovering drops of molten metal by cooling.

When a molten metal is poured into the reservoir **4** continuously or semicontinuously to keep the level of the melt constant, it runs through the sprue runner **5** which is provided with a heating and heat preserving means and pours out almost constantly from the tip of the nozzle **6**.

On one hand, the compressed air to serve as the medium for atomization is supplied from the blast pipe **10** and follows the course shown in the drawing.

This air of relatively low pressure of 0.5–1.5 kilogrammes per square centimetre with an obtuse ejection angle α forms a relatively constant flow without eddying, and meets the molten metal while forming an atmosphere of a reduced pressure caused by sudden expansion of the compressed air at the ejecting zone corresponding to lines connecting respectively the tip points T and Ta of the nozzle **6** with the inner edge point R and Ra of the ejecting opening **17** of the cap **13**, whereby a substantially conical active zone depending on variation of the air pressure is established, thus causing atomization of the molten metal while tearing off said molten metal.

The above-mentioned angle α corresponds to the angle formed between a line, which passes through the central point of a line R–T and the central points of lines drawn parallel to said line R–T in the air chamber of the cap **13**, and another line passing through the central point of a line R–T and the central points of lines drawn in parallel to said line Ra–Ta in the air chamber of the cap **13**.

For the establishment of the appropriate atomization, it is necessary to increase the energy of the compressed air up to a value adapted to carry out pulverization with respect to a certain pressure adapted to the above-mentioned free dropping state and when said energy and pressure are balanced, a well-regulated active zone is first established in a continuous manner. The atomizing condition adapted to said pulverization can be easily satisfied when the tip of both nozzle is positioned within the range of protruding length p of the nozzle. This length p corresponds to the distance between a line (T–Ta) and a vertical line passing through the cross-point q between a line m–n perpendicular to the line Lα and passing the point R and the outer slant line of the nozzle end. Now, when the tip of the nozzle is affixed at a position within the distance range of p, pressure of the compressed air should be raised so as to be balanced with the pressure of the molten metal flowing out of the nozzle in accordance with said pressure of the molten metal which is proportional to metal head and specific gravity thereof in the crucible, whereby the frictional force at the active zone is increased in proportion to said raised pressure of the compressed air, thus increasing the fine powder producing yield. In this case, when the above mentioned balancing is established by maintaining the metal head in the crucible at a certain value, an active zone of a certain size is established.

However, the configuration of said zone is narrowered gradually in accordance with gradual increase of the air pressure, whereby said zone can be made to be minimum at the tip of the nozzle, that is, to be small corresponding to the state just prior to the state in which flowing-up phenomenon of the molten metal is established by the action of negative air pressure adapted to disturb flowing-out of the molten metal, thus increasing the frictional force up to a maximum and causing maximum production yield at a certain position of the tip of the nozzle.

On the other hand, if it be assumed that the tip of the nozzle is transferred within the range of the distance P from the O line thereof ($P=O$), the width of the ejecting ring zone is gradually decreased with gradual increase of the distance P and the obtuse ejection angle increases gradually, whereby the pressure of the blowing air is increased under the condition such that the blowing amount of the air is constant. This increase of the pressure of the blowing air causes an increase of the fine

powder production yield, but a decrease of the flowing-out velocity of the molten metal. Thus, said velocity of the molten metal becomes minimum and said production yield becomes maximum at the position of the nozzle tip where it is just to the rear (by 1.0 millimeter) of the line (R–Ra). When the nozzle tip advances outwards from said position, the ejection angle α suddenly becomes an acute angle even when said advance is very slight, whereby the active zone extends lengthwise in the atomization direction so as to increase suddenly the flowing-out amount of the molten metal, thus causing production of coarse granular powders of a large amount. If the nozzle tip is further advanced than the said advance, a large amount of plate-shaped particles is produced.

On the contrary, if the nozzle tip is retracted from the O line ($P=O$), the width of the ejecting annulus becomes excessively large so as to cause turbulence of the air stream, whereby the molten metal is sprayed and adhered onto the inner surface of the cap. Of course besides the above-mentioned powdering action, the higher the temperature of the molten metal is, the lower the viscosity and surface tension of the molten metal, whereby the fine powder production yield is increased, and moreover, the larger the specific gravity of the molten metal, the lower the metal head, thus increasing the fine powder production yield.

It seems that in general, in the case of atomizing any molten metal, shearing action is produced by the friction occurring between the surface of the molten metal blown out of the nozzle tip and the compressed air stream, whereby the stream of the molten metal is torn off into fine powder. Since the action of this pulverizing mechanism depends upon physical properties, such as viscosity, surface tension, density and the like of the molten metal, said action is affected by various factors such as temperature of the molten metal, cross-sectional area of the stream of the molten metal, pressure and flowing-out velocity of the molten metal based on the metal head in the crucible, the distance the nozzle protrudes, condition of the compressed air and the like. Particularly, configuration, opening diameter and width of the ejecting annulus, configuration ejecting angles of the nozzles for atomizing molten metal and compressed air, configuration of the nozzle box, moving condition of the compressed air in the air chamber, and entering direction, pressure and velocity of the compressed air in the nozzle box are factors capable of causing variation of the condition of the compressed air.

The inventor of the present invention has found, as a result of various studies relating to the above-mentioned conditions by utilizing various atomizing devices, that adoption of physically and chemically robust material for the inner tube **7** of the molten metal atomizing nozzle, adoption of such configuration, cross-sectional dimension of said nozzle and movable length of the tip of said nozzle as disclosed in this specification, adoption of an obtuse ejecting angle, adoption of such sectional dimension of the air chamber **16** in the cap, and opening diameter and width of the ejecting annulus **17** as disclosed in this specification, said opening diameter and width being matched with said sectional diameter of said air chamber, are the most appropriate conditions for increasing fine powder production yield and production rate on an industrial scale.

In general, it is very difficult to obtain at the same time good results both in yield for recovering "fines" and in the production rate with a relatively low air pressure. Therefore, when it is required to produce "fines" of less than 43 microns on a large scale with a yield of more than 80%, the cross-sectional area of the molten metal nozzle must be made as small as possible, and this fact is apparently unfavourable for the atomization mechanism in order to increase the rate of production. If the conventional atomizing device were operated with the configuration and angle of ejection of the air ejecting nozzle, said configuration causing rotation of the ejected air stream and said angle being an acute angle (in general, 60°) differring from those of the device of this invention, it is difficult to respond quickly to the variations in the ejected air stream and ejection angle, that is, to the reduction in the powdering efficiency, said variations being caused by wear of the tip of the molten metal nozzle by melting, which has long been considered to be the most difficult problem in this process of atomization. Consequently, continuous operation over a long time would certainly end in failure and economical mass-production on an industrial scale by the conventional method would be handicapped.

The present inventor has discovered the following facts from various investigations in order to make economical mass-production possible by overcoming the many difficulties mentioned above. That is, when the quantity and pressure of the compressed air for atomizing molten metal are respectively limited to the maximum ranges of 8 cubic meters per minute and 1.5 kilograms per square centimetre, and the cross-sectional area of the cap air chamber **16** and its air containing capacity are increased in a cup form while maintaining a balance with its nozzle box air chambers **12A** and **12B** formed by doubled rings, the air stream is caused to contact the molten metal at an obtuse ejection angle α as a steady current of constant flow which does not cause vortices, although said air stream meets the surface of molten metal stream near the active zone, whereby a large frictional force capable of causing a strong contact of the air stream with the molten metal is produced, thus applying a vigorous shearing action to the molten metal. On the contrary, in the case of contact at an acute angle as in the case of a swirling-type atomizer producing a rotating jet in the direction of 60 degrees, a shearing action as vigorous as in the former case can never be produced without using compressed air of higher pressure overcoming the force consisting of said frictional force and an additional friction force caused by rotation of the ejected stream.

Thus, in the former case, the energy of the compressed air is concentratedly consumed most effectively within a very short distance of the active zone, and the molten metal is drawn in the direction of the air stream which rapidly expands and reduces its pressure in this zone, and is ejected and atomized forming the characteristic conical active zone.

This invention is based on the discovery of the fact that the ejected air stream is in the best condition when it takes such a conical form, which makes it possible to utilize an unprecedentedly large cross-sectional area of the inner tube **7** of molten metal nozzle **6** in accordance with the required air volume. For example, in the case of zinc, lead, cadmium and the like, when the diameter of the inner tube of the nozzle and the metal head are respectively selected to be 9 millimeters and 620 millimeters, outflow-velocity of the melt can be made to be above 230 kilogrammes per hour while maintaining the production yield of fine particles of a size below 43 microns at a value above 80%. Moreover, it has become possible to prepare profitably "fines" which contain principally particles of 30–40 microns size accompanied by some "superfines." Furthermore, when the diameter of the inner tube of the nozzle and the metal head are respectively selected to be 15 millimeters and 690 millimeters, mass-production has become possible at an outflow-velocity of the melt above 280 kilogrammes per hour.

Thus, this invention makes it possible to increase readily the production rate by the use of compressed air of relatively low pressure without losing high yield and high efficiency by improving the atomizing device on the basis of the discovery that the condition of the ejected air stream is greatly influenced by the constructions of the air chamber **16**, nozzle **6** and its inner tube **7**.

According to this invention, it is possible to obtain

plenty of fine metal powder containing only slight oxide and super fine powder and to adjust particle size distribution of atomized powder within a relatively wide range.

Furthermore, because of the use of an inner tube of the molten metal nozzle made of special refractory materials (which include silicates, alumina, silicon carbide, zirconia and the like), the life of the molten metal nozzle can be increased by several times in comparison with that of a nozzle made of mild steel, and high purity powder of a metal such as zinc, aluminum or the like, which is free of iron as an impurity can be produced easily and stably without any trouble.

For example, it is possible to prepare, from electrolytic zinc, high purity zinc powder containing more than 99.98% of pure zinc, less than 1.5% of zinc oxide, less than 0.0005% of cadmium, less than 0.0010% of iron, less than 0.0030% of lead, and moreover, it is possible to maintain over a long period a high production rate on an industrial scale of more than 250 kilogrammes per hour with a yield of more than 30% of "fines" of less than 43 microns. The peak of the particle size distribution of metal powder prepared by the novel atomizing device of this invention resides at about 30 or 40 microns, accompanied by "super fines" of less than 10 microns which can be recovered by utilizing a pneumatic classifier. In addition, it is also possible to prepare metal powder of any desired particle size distribution by the combined use of sieves and a mixer.

In order to indicate still more fully the nature and utility of the present invention, the following examples of typical process conditions and corresponding results are set forth, it being understood that these examples are presented as illustrative only, and that they are not intended to limit the scope of the invention.

EXAMPLE 1

(A) Condition of atomization

Metal to be atomized	Metal zinc.
Type	Horizontal atomizing method.
Cap:	
Width of the ejecting annulus.	6.0 mm.
Diameter of the ejecting opening.	24.0 mm.
Length of nozzle protrusion	1.5 mm.
Angle of ejection	About 120°.
Pressure of compressed air	0.9–1.0 kilogrammes per square centimetres.
Metal bath nozzle:	
Diameter of the inner tube	12.0 mm.
Material composing the inner tube of the nozzle.	Siliceous refractory material.
Maximum height of the molten metal in the crucible.	620 mm.
Minimum height of the molten metal in the crucible.	570 mm.
Range of regulation of the height of the molten metal.	50 mm.
Temperature of the molten metal at the time of atomization.	About 550° C.
Method of cooling	Natural air cooling.

(B) Results of operation

Quantity of product per hour ____ 260 kilogrammes per hour.

Production yield of the powder according to particle size:

	Weight percent
Smaller than 76μ	90.5
Smaller than 43μ	75.6

(C) Quality of the zinc powder produced

Composition of product according to particle size:

	Percent					
	T. Zn	M. Zn	O. Zn	Fe	Cd	Pb
Particle size:						
Smaller than 76μ	99.98	98.57	1.41	0.0010	0.0005	0.0030
Smaller than 43μ	99.98	98.28	1.70	0.0010	0.0005	0.0030

EXAMPLE 2

(A) Condition of atomization

Metal to be atomized	Metal zinc.
Type	Horizontal atomizing method.
Cap:	
Width of the ejecting annulus	5.0 mm.
Diameter of the ejecting opening.	23.0 mm.
Length of nozzle protrusion	5.0 mm.
Angle of ejection	About 125°.
Pressure of compressed air	1.5 kilogrammes per square centimetres.
Metal bath nozzle:	
Diameter of the inner tube	9.0 mm.
Material composing the inner tube of the the nozzle.	Siliceous refractory material.
Maximum height of the molten metal in the crucible.	620 mm.
Minimum height of the molten metal in the crucible.	590 mm.
Range of regulation of the height of the molten metal.	30 mm.
Temperature of the molten metal at the time of atomization.	About 550° C.
Method of cooling	Natural air cooling.

(B) Results of operation

Quantity of product per hour ____ 230 kilogrammes per hour.

Production yield of the powder according to particle size:

	Weight percent
Smaller than 76μ	95.0
Smaller than 43μ	88.0

(C) Quality of the zinc powder produced

Approximately the same as in Example 1.

EXAMPLE 3

The same tests as the Examples 1 and 2 were carried out in connection with the lead, tin, cadmium, antimony, bismuth, and aluminum under the same conditions as in the case of the Examples 1 and 2, and substantially similar results were obtained.

What we claim is:

1. An apparatus for preparing high purity fine powder of a metal selected from the group consisting of zinc, lead, tin, cadmium, antimony, bismuth, aluminum and similar metals having a relatively low melting point, which apparatus comprises a crucible of refractory material and having a reservoir chamber for containing molten metal therein, a heating and heat preserving means surrounding said crucible, a uniform diameter nozzle connected with the lower end of said reservoir chamber and below the level thereof for ejecting the molten metal and having the external end surface tapered inwardly, a nozzle box supporting said nozzle, means supplying air under pressure of at most 1.5 kilograms per square centimeter to eject the molten metal out of the opening of said nozzle, said means comprising an apertured cap which is adjustably attached to said nozzle box in front of said nozzle

for movement in the axial direction of said nozzle to vary the position of the tip of said nozzle relative to said cap aperture, said cap and nozzle box defining a ring-shaped cap air chamber inside of said cap, a ring-shaped inner air chamber in said nozzle box and opening into said cap air chamber, an outer ring-shaped air chamber around said nozzle box and opening into said inner ring-shaped chamber, an air blast pipe connected to said means to supply air to said air chamber, and a dust chamber in front of said cap adapted to recover the powdered molten metal, said nozzle being spaced inwardly of said cap and having an air flow ejecting angle which is the cone angle of a cone generated by a line rotating around the nozzle axis and bisecting a connecting line between the edge of said cap aperture and the edge of said nozzle and series of lines across said air chamber and parallel to said connecting line, said cone angle being an obtuse angle, and the end of said nozzle lying between the flat plane across the cap aperture at the inner surface of the cap and a flat

plane perpendicular to the nozzle axis through the points on the tapered external end surface of the nozzle intersected by a further cone through the said cap aperture at said inner surface and perpendicular to said first mentioned cone.

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