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(54) **Title:** HIGH MELTING POINT METAL OR ALLOY POWDERS ATOMIZATION MANUFACTURING PROCESSES

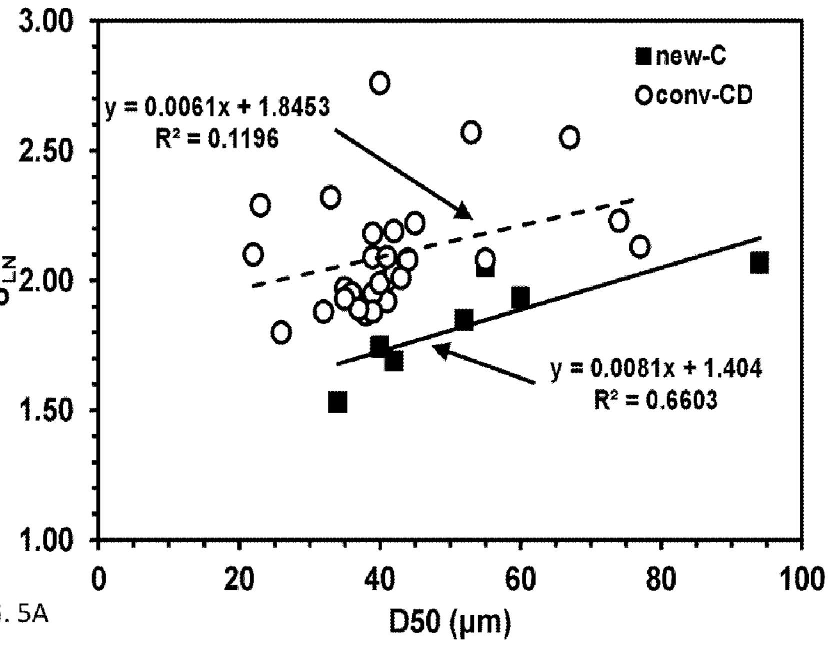

FIG. 5A

(57) **Abstract:** There are provided high melting point metal or alloy powder atomization manufacturing processes comprising providing a melt of the high melting point metal or alloy through a feed tube; diverting the melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt; directing the diverted melt to an atomization area; and providing at least one atomization gas stream to the atomization area. The atomization process can be carried out in the presence of water within an atomization chamber used for the atomization process.

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HIGH MELTING POINT METAL OR ALLOY POWDERS ATOMIZATION MANUFACTURING PROCESSES

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] The present application claims the benefit of priority from U.S. provisional application no. 62/631,286 filed on February 15, 2018. This document is hereby incorporated by reference in its entirety.

FIELD OF THE DISCLOSURE

[0002] The field of the disclosure pertains to the production of fine metallic powders for application in the electronic industry, metal injection forming, thermal spraying, thermal spray welding, 3D printing and catalyst materials.

BACKGROUND OF THE DISCLOSURE

[0003] Many new materials with outstanding physical and chemical properties can be synthesized but remain difficult to produce economically at industrial scale by conventional methods (casting/machining). Some of these materials are synthesized or deposited by alternative techniques such metal injection forming, 3D printing, thermal spaying and other techniques requiring powders with specific size distribution, sphericity and physical properties. Electronic devices and components have also been significantly reduced in size and they also require fine metallic powders in formulations for solder paste or ink used to apply conductive materials containing metallic powders. In brief technology is advancing and in order to enable more innovative bulk materials, coating, conductive layers, metallization and metal forming applications, metallic powders of relatively fine size distribution and of relatively tight size distribution are in increasing demand. Some other applications of fine powders are also seen in catalytical materials where selected precious metals or metals having multiple oxidation states are also used. In this later case, fine metallic powders can be produced and dispersed on a media to serve together as a catalytical material. It is not uncommon to have required or requested particle size distribution mostly under 50 and even under 20 microns for such applications.

[0004] There are multiple other applications for fine metallic powders, such as metal injection forming, thermal spraying, thermal spray welding, 3D printing and many more.

[0005] Conventional techniques (atomization, centrifugal disintegration, water atomization...) can produce fine powders, butsmaller particle size, low standard deviation on size distribution and the spherical shape of the particles are difficult to achieve from metals or alloys with these techniques. This often leads to a low recovery of the produced powder in a defined size fraction from these technologies.

SUMMARY OF THE DISCLOSURE

[0006] The present disclosure describes a new production process for metallic powders having high melting points. This process produces fine spherical powders with a small standard deviation on the particle diameter.

[0007] In a first aspect, there is provided a high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt;
directing the diverted melt to an atomization area; and
providing at least one atomization gas stream to the atomization area,

[0008] The atomization process may be being carried out in the presence of water within an atomization chamber used for said atomization process.

[0009] In a second aspect, there is provided a high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
delivering said melt through a diverter to an atomization area;
providing at least one atomization gas stream to the atomization area;
delivering water to an atomization chamber used for said atomization process, wherein, prior to being delivered to the atomization area, the melt is diverted in the diverter at a diverting angle with respect to a central axis of the feed tube.

[0010] In a third aspect, there is provided a a high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube; directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 50 microns with geometric standard deviation of lower than about 2.2.

[0011] In a fourth aspect, there is provided a high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
optionally diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain an optionally diverted melt;
directing the optionally diverted melt to an atomization area; and
providing at least one atomization gas stream having a velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000- cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder particle sizes having geometric standard deviation of lower than about 2.2.

BRIEF DESCRIPTION OF DRAWINGS

[0012] For a better understanding of the various embodiments described herein, and to show more clearly how these various embodiments may be carried into effect, reference will be made, by way of example, to the accompanying drawings which show at least one example embodiment, and in which:

[0013] Figure 1 is a block diagram illustrating steps involved in the atomization process, in accordance with at least one embodiment;

[0014] Figure 2 illustrates a schematic side view of an atomization nozzle with a feed tube with a diverting channel to provide the melt in the atomization area, in accordance with at least one embodiment;

[0015] Figure 3 illustrates a perspective view of the atomization chamber showing tangential gas entries on the gas inlet, in accordance with at least one embodiment;

[0016] Figures 4A and 4B illustrate scanning electron microscope (SEM) pictures of the powder obtained in Example 2, wherein Figure 4A refers to a Type 5 powder (15-25 µm) and Figure 4B refers to a the proportion of the powder under 7 µm; and

[0017] Figures 5A and 5B illustrate the benefit of the new atomization technology (new-C) compared to a reference conventional “Convergent-Divergent (conv-CD)” atomizer, wherein Figure 5A indicates a lower standard deviation in size distribution for the new technology and Figure 5B indicates a higher yield inside a prescribed particle size range.

DESCRIPTION OF VARIOUS EMBODIMENTS

[0018] The following examples are provided in a non-limitative manner.

[0019] The expression “high melting point metal” as used herein refers to a metal having a melting point temperature of about 500° Celsius to about 1800° Celsius.

[0020] The expression “high melting point alloy” as used herein refers to an alloy having a liquidus temperature of about 500° Celsius to about 1800° Celsius.

[0021] Terms of degree such as “about” and “approximately” as used herein mean a reasonable amount of deviation of the modified term such that the end result is not significantly changed. These terms of degree should be construed as including a deviation of at least ±5% or at least ±10% of the modified term if this deviation would not negate the meaning of the word it modifies.

[0022] In understanding the scope of the present disclosure, the term “comprising” and its derivatives, as used herein, are intended to be open ended terms that specify the presence of the stated features, elements, components, groups, integers, and/or steps, but do not exclude the presence of other unstated features, elements, components, groups, integers and/or steps. The foregoing also applies to words having similar meanings such as the terms, “including”, “having” and their derivatives. The term “consisting” and its derivatives, as used herein, are intended to be closed terms that specify the presence of the stated features, elements, components, groups, integers, and/or steps, but exclude the presence of other unstated features, elements, components, groups, integers and/or steps. The term “consisting essentially of”, as used herein, is intended to specify the presence of the stated features, elements, components, groups, integers, and/or steps as well as those that do not materially affect the basic and novel characteristic(s) of features, elements, components, groups, integers, and/or steps.

[0023] In the production of fine metallic powders, there are several parameters that can affect product quality. Some of the parameters used to characterize powders may include average size distribution, standard deviation of the size distribution, proportion of coarser particles and finer particles over/under predefined sizes, sphericity of the powder, level of metallic impurities and oxygen level.

[0024] In at least one embodiment, the diverting angle (90-Beta) may be about 30 to about 70 degrees.

[0025] In at least one embodiment, the diverting angle may be about 10 to about 90 degrees.

[0026] In at least one embodiment, an angle formed between the atomization gas and the melt may be about 10 to about 90 degrees.

[0027] In at least one embodiment, an angle formed between the atomization gas and the melt may be about 40 to about 90 degrees.

[0028] In at least one embodiment, the process may comprise providing a high melting point metal.

[0029] In at least one embodiment, the high melting point metal may have a melting point of about 500° Celsius to about 1800° Celsius.

[0030] In at least one embodiment, a ratio of the atomization gas to the high melting point metal in the atomization area may be about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal to atomize.

[0031] In at least one embodiment, a ratio of the atomization gas to the high melting point metal in the atomization area may be about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize.

[0032] In at least one embodiment, the high melting point metal may be an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au.

[0033] .In at least one embodiment, the high melting point metal may be an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

[0034] In at least one embodiment, the high melting point metal is Cu.

[0035] In at least one embodiment, the high melting point metal is Sn.

[0036] In at least one embodiment, the process may include providing a high melting point alloy.

[0037] In at least one embodiment, the high melting point alloy may have a liquidus of about 500° Celsius to about 1800° Celsius.

[0038] In at least one embodiment, the high melting point alloy may have a liquidus of about 500° Celsius to about 1500° Celsius.

[0039] In at least one embodiment, a ratio of atomization gas to the high melting point alloy may be about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal.

[0040] In at least one embodiment, a ratio of atomization gas to the high melting point alloy may be about 5000 to about 40 000 cm^3 of gas per cm^3 of metal.

[0041] In at least one embodiment, the high meting point alloy may include at least one element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au.

[0042] In at least one embodiment, the high meting point alloy may include at least one element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

[0043] In at least one embodiment, the high meting point alloy comprises Cu.

[0044] In at least one embodiment, the high meting point alloy comprises Sn.

[0045] In at least one embodiment, the high meting point alloy comprises Cu and Sn.

[0046] In at least one embodiment, the high melting point allow consists essentially of Cu and Sn.

[0047] In at least one embodiment, the high melting point allow consists of Cu and Sn.

[0048] In at least one embodiment, the atomization gas stream may have a velocity of about 300 m/s to about 700 m/s.

[0049] In at least one embodiment, the atomization gas stream may have a velocity of about 450 m/s to about 600 m/s.

[0050] In at least one embodiment, the atomization gas stream may have a supersonic speed.

[0051] In at least one embodiment, the atomization gas may be delivered to an atomization head through at least one gas inlet oriented in a non-perpendicular way with respect to the atomization head, the gas inlet providing a swirl movement in the atomization head prior to the gas exit.

[0052] In at least one embodiment, at least two gas injectors may be offset versus the central axis of the feed tube, creating a dynamic rotational effect around the central axis in the atomization area.

[0053] In at least one embodiment, the process may thereby provide a distribution of powder particle sizes with geometric standard deviation of lower than or about 2.2.

[0054] In at least one embodiment, the process may thereby provide a distribution of powder particle sizes with geometric standard deviation of about 1.5 to about 2.0.

[0055] In at least one embodiment, the atomization chamber may comprise about 0 to about 20% of oxygen.

[0056] In at least one embodiment, the water may comprise at least one additive to reduce the redox potential of the water.

[0057] In at least one embodiment, the redox potential of the water has been reduced prior to the atomization.

[0058] In at least one embodiment, the temperature of the water used in the atomization chamber is lowered so as to reduce the powders oxidation in the atomization process

[0059] In at least one embodiment, the process may thereby provide powder average particle size of about 10 microns to about 50 microns in diameter.

[0060] In at least one embodiment, the melt of said high melting point metal may be diverted through at least one melt diverting channel and the diverting angle may be formed between the central axis of the feed tube and the at least one melt diverting channel.

[0061] In at least one embodiment, the alloy melt may be diverted through at least two melt diverting channels and the diverting angle may be formed between the central axis of the feed tube and the at least two melt diverting channels.

[0062] In at least one embodiment, at least one jet of water may be sprayed into the atomization chamber.

[0063] In at least one embodiment, the at least one jet of water may be sprayed on at least one wall of the atomization chamber.

[0064] In at least one embodiment, the process may thereby provide a powder having an average particle size of less than about 50 microns.

[0065] In at least one embodiment, the process may thereby provide a powder having an average particle size of less than about 35 microns.

[0066] In at least one embodiment, the produced powder may be vacuum dried to avoid powders oxidation.

[0067] In at least one embodiment, the produced powder may be washed with an organic solvent to remove most of the water prior of the drying stage.

[0068] In a fifth aspect, an atomization device for manufacturing high melting point metal or alloy powder is provided. The device may include a feed tube for providing a melt of said high melting point metal or alloy; a diverter, in fluid flow communication with said feed tube, for diverting the melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt, and to directing the diverted melt to an atomization area of the atomization device; at least one atomization gas injector for providing at least one atomization gas stream to the atomization area located inside the atomization chamber; and at least one water inlet for providing water within an atomization chamber of said atomization device.

[0069] In at least one embodiment, the diverter may comprise a melt diverting conduit, the diverting conduit being oriented at a diverting angle with respect to a central axis of the feed tube.

[0070] In at least one embodiment, the diverter may comprise at least two melt diverting conduits, each of the at least two melt diverting conduits being oriented at a diverting angle with respect to a central axis of the feed tube.

[0071] In at least one embodiment, the device may comprise at least one gas inlet, the at least one gas inlet being non perpendicular to the atomization head as to provide a swirl movement in the atomization head and a dynamic rotational movement in the atomization area and the atomization chamber.

[0072] In at least one embodiment, at least one non perpendicular gas inlets may create a circular flow in the atomization head leading to a dynamic rotational movement of the gas in the atomization area and the atomization chamber.

[0073] In at least one embodiment, at least two gas inlets may be non perpendicular to the atomization head creating a swirling effect in the atomization head and a dynamic rotational effect in the atomization area and the atomization chamber.

[0074] In at least one embodiment, the at least one water inlet may be located inside the atomization chamber.

[0075] In at least one embodiment, the at least one water inlet may be suitable for providing water for cooling said powder.

[0076] In at least one embodiment, the at least one water inlet may be suitable for providing water for transporting said powder to the sieving/drying area.

[0077] In at least one embodiment, the at least one water inlet may be suitable for providing water for facilitating sorting/sieving of said powder.

[0078] The described process is based on a known concept, atomization, but with several specific improvements. These improvements include changes to the atomization head operating parameters, to the atomization chamber configuration and to the means of post processing of the powder (collection, sieving and drying) prior of packing the final product. The process is designed to reach advanced product quality and high process performances.

[0079] Figure 1 shows a block diagram 100 of apparatus and steps involved in the atomization process, in accordance with at least one embodiment. Figure 1 shows a melting furnace 102, the atomization nozzle 200, the atomization chamber 108, a powder collection system 112 and a sieving system 114.

[0080] Most highmelting point alloys and/or high melting point metals produced with this process are sensitive to oxidation, hence the atomization gas may advantageously be an inert gas. The system may be generally maintained in near inert conditions with oxygen levels much under 21% in the atomization chamber 108. In order to save operating costs, this gas may be purified/recycled in the process.

[0081] In at least one embodiment, the atomization manufacturing process may be carried out by the atomization nozzle 200 where the atomization gas meets with a metal flow in specific conditions described herein. Shown at Figure 1 also shows a schematic side view of the atomization nozzle 200, where the molten metal may contact the atomization gas in the atomization zone.

[0082] Once the metal has been solidified in fine powders, it is sieved and packed.

[0083] Referring to Figure 1, some water may be added in the atomization chamber 108 through the side nozzles 120 and 122 to help collecting the powder and to bring the liquid mixture of the powder and water to the sieving area 114. These water addition side nozzles 120 and 122 may be oriented towards the atomization chamber walls or may be located in the atomization area to help cooling of the powder and to avoid adhesion/deformation of the particles on the atomization chamber walls. Water can also be added to ease powders collection and sieving. The produced powders may then be sieved and dried. After collection of the bulk of the powder, from the liquid stream, the bulk of the powder passes into filter presses 116 to recover all remaining powders in suspension prior to water recycling/disposal.

[0084] The size distribution of the powder produced during the optimization manufacturing process can be affected by the speed at which the atomization gas hits the metal. In this regards, higher velocity of the atomization gas leads to lower size distributions of the powder. If the atomization nozzle 200 is not designed properly, a smaller portion of the metal will be meeting the atomization gas in the required conditions (atomization gas velocity and volume) and larger variations in size and shape of the produced powder may be observed. The intimate contact between the high melting point metal/alloy and the atomization gas is also important.

[0085] Figure 2 illustrates a schematic side view of an atomization nozzle 200. The atomization nozzle 200 has a feed tube 210 with a diverting channel 216 to provide the melt in the atomization area 230.

[0086] As shown at Figure 2, the atomization nozzle described herein comprises a feed tube 210 located between the melting furnace 102 and the atomization area 230 which is equipped with a diverter 216 (also called herein as a diverting channel 216). The role of this diverter 216 is to provide a better contact between the metal and the gas in the atomization zone 230.

[0087] The metal being hit by the atomization gas stream at a sheer angle Gamma defined as Gamma = 90-Beta+Alpha. This approach provides additional parameters for improvement of the atomization process: Beta angle, as well as diameter and number of diverter channels 216.

[0088] In at least one embodiment, the metal may be diverted in the atomization area 230 with the Beta angle being about 20 to about 60 degrees. For example, the atomization gas may be provided to the atomization area 230 at an Alpha angle of about 20 to about 35 degrees.

[0089] For example, if the sheer angle Gamma is about 90 degrees, or at least about 60 to about 120^{o}, the atomization may be improved, by an enhanced gas to metal contact and higher sheer energy

[0090] The melt diverting angle is also defined herein as 90-Beta.

[0091] The Alpha angle, at which the atomization gas may be provided with respect to the feed tube 210, may also have other limitations. For example, if angle Alpha is more than 60 degrees, a close to direct projection of the atomization gas on the atomization chamber walls may require larger atomization chamber diameters.

[0092] For example, Alpha angle may be as low as about 20 to about 45°.

[0093] For example, Alpha angle may be less than about 20 to about 45°.

[0094] In at least one embodiment, the Alpha angle may be between about 0 to about 90°; about 10 to about 50 °; about 15 to about 50 °; about 20 to about 50 °.

[0095] In at least one embodiment, the Alpha angle may be about 20 to about 45° where 2 Alpha may be about 40° to about 90°). In at least one embodiment, the Alpha angle may be about 20 to about 40 °; about 30 to about 45 °.

[0096] Once the metal/alloy is hit by the atomization gas, small particles are formed. Collisions between those particles may produce satellites (many particles connected together) and may also produce of non-spherical metallic particles, both of which need to be avoided and/or reduced or prevented. This may be partially done by modifying Alpha and Beta angles, as well as the average atomization gas velocity and the dispersion factor.

[0097] In order to avoid collision prior to solidification, the density of particles in the atomization gas need to be controlled in an appropriate range. For example, if one cubic centimeter (cc) of metal is atomized in 10 microns diameter spherical particles in $1M^3$ of atomization gas, the density of particles in the plume is 1,9 Millions/M^3. The use of $5M^3$ of gas per cubic centimeter of metal would reduce this density by a factor 5. So an optimal range of gas volume per metal volume is critical to avoid collisions and also to provide the sheer energy to pulverize the metal in small dropplets and also providing proper heat exchange mechanism to solidify the dropplets rapidly. The use of 5000 to 40000 cm^3 of atomization gas per cubic centimeter of metal/alloy was found appropriate for the production of fine powders (under 50 microns) of high melting point metals/alloys.

[0098] Described herein are the velocity and the dispersion as being critical factors influencing the atomization results (fineness and avoidance of satellites and non atomized metal/alloys).

[0099] In at least one embodiment, the atomization device 150 may include at least one non-perpendicular atomization gas inlet 214 with respect to the gas feed tube axis 212, leading to a rotational movement of the atomization gas stream 240 in the atomization head 222. In an extreme example embodiment described below, the gas inlets 214 enter in the atomization head tangentially.

[00100] Figure 3 illustrates a perspective view of the atomization chamber 300 showing tangential gas inlets 311 and 314, in accordance with at least one embodiment. This design may allow for an asymmetric atomization plume in dynamic rotation around a central axis 312. This configuration of the atomization gas inlets may provide an improved particle size distribution compared to an atomizer with perpendicular gas entries with respect to the feed tube central axis 312.

[00101] Some high melting point metals/alloys are difficult to solidify. . If some particles touch the walls of the atomization chamber 108 and are still partially molten or close to their melting points, they can be significantly deformed to reach a flake-type morphology, agglomerate and form non spherical particles or satellites (several particles connected together). In order to reduce these phenomena, the described atomization technology can use water as a cooling media. The water may be injected in direction of the atomization chamber walls to provide a film of water carrying the produced powder. The film of water may ensure that metallic powders or metal droplets are cooled at a sufficient temperature to reduce or avoid the sticking particles, satellites and/or deformed particles. The water, in some cases, may provide a controlled level of surface oxidation, which may also contribute to have a free flowing powder with an acceptable level of oxygen in the final product.

[00102] For example, adding water in the atomization chamber (on walls, in the upper part of the atomization chamber or at the bottom of the atomization chamber) may also improve material classification. Due to electrostatic forces being enhanced between fine particles, it is sometimes hard to separate particles if dry sieving is used. Some high melting point alloys/metals powders tend to agglomerate together for many reasons. For example, sintering or sticking of the particles and also for electrostatic reasons as mentioned above. While the exact reason for agglomeration is not fully known for all high melting point/alloys produced, there is a benefit for a wet sieving system for several alloys.

[00103] The use of water in this process may be counterintuitive, as some alloying elements/metals may theoretically oxidize in presence of water. Many elements, such as Fe, for example, may even reduce water in absence of dissolved oxygen in water. For example, when a low oxygen level is maintained in the atomization chamber, the oxidation of the produced powder may be inside acceptable levels. In addition of controlling the oxygen in the atmosphere of the atomization chamber, the redox potential and the temperature of the water used in the process (for the atomization chamber and for the sieving) may be controlled, leading to a reduced kinetic of oxidation.

[00104] Some metallic powders, made of high melting point metals/alloy, may need a controlled oxidation to remain free flowing in the final product. Optionally, oxygen peroxide or other hydrometallurgical oxidants may be addedin the water to allow a controlled level of oxidation. Alternatively, the powder may be left in water at a controlled temperature for a given period of time (with or without steering) to allow for a controlled oxidation of the powder.

[00105] While a controlled oxidation is beneficial for some products, overly high levels may be generally detrimental. Optionally, the redox of the incoming water may be lowered to limit oxidation. This can be done by adding additives in the water used in the atomization process (in the chamber or in the sieving system) to reduce the level of oxygen in the final product. Additives can be reducing agents, like organic additives, such as ethanol, methanol, formic acid, acetic acid, methane sulfonic or inorganic reductants. Redox potential in water may also be reduced by diverse other means, including but not limited to electrochemicals system to treat incoming water, reduction of temperature, filter with reactive metal powders.

[00106] In at least one embodiment, the dissolve oxygen in the incoming water may be controlled to limit oxidation in the product. In at least one embodiment, the metal film on the powder may be reduced by dissolution with mild acid (HCl, organic acids, etc.). These may be added in the water to reduce the oxide film formed at the powder surface.

[00107] One of the final production steps of the process is to dry the powder. This step can be performed atmospherically, under vacuum or in an inert gas. Vacuum allows the drying process to operate at a lower temperature, hence reducing potential oxidation

with the water. Optionally, prior of the drying stage, water can be displaced from the powder using an organic solvent in which water is soluble. For example ethanol and methanol. After the water has been removed, the powder containing some residual organic liquid can be dried to produce a final product with low level of oxygen.

[00108] In at least one embodiment, a high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt; directing the diverted melt to an atomization area; and providing at least one atomization gas stream to the atomization area. Said atomization process being carried out in the presence of water within an atomization chamber used for said atomization process.

[00109] In at least one embodiment, the high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; delivering said melt through a diverter to an atomization area; providing at least one atomization gas stream to the atomization area; delivering water to an atomization chamber used for said atomization process, wherein, prior to being delivered to the atomization area, the melt is diverted in the diverter at a diverting angle with respect to a central axis of the feed tube.

[00110] In at least one embodiment, the high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 50 microns with geometric standard deviation of lower than about 2.0. In at least one embodiment, the high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area,

wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 435 microns with geometric standard deviation of lower than about 2.2.

[00111] In at least one embodiment, the high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 350 microns with geometric standard deviation of lower than about 2.0. In at least one embodiment, the high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 50 microns with geometric standard deviation of lower than about 2.2.

[00112] A high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high melting point metal or alloy through a feed tube; optionally diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain an optionally diverted melt; directing the optionally diverted melt to an atomization area; and providing at least one atomization gas stream having a velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000- cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder particle sizes having geometric standard deviation of lower than about 2.0. A high melting point metal or alloy powder atomization manufacturing process may include providing a melt of said high

melting point metal or alloy through a feed tube; optionally diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain an optionally diverted melt; directing the optionally diverted melt to an atomization area; and providing at least one atomization gas stream having a velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000- cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder particle sizes having geometric standard deviation of lower than about 2.2.

[00113] For example, the diverting angle (90-Beta) may be about 30 to about 70 degrees.

[00114] For example, the diverting angle may be about 10 to about 90 degrees.

[00115] For example, an angle formed between the atomization gas and the melt may be about 10 to about 90 degrees. For example, an angle formed between the atomization gas and the melt may be about 40 to about 90 degrees.

[00116] In at least one embodiment, the process may also include providing a high melting point metal.

[00117] In at least one embodiment, the high melting point metal may have a melting point of about 500° Celsius to about 1800° Celsius.

[00118] In at least one embodiment, a ratio of the atomization gas to the high melting point metal in the atomization area may be about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal to atomize. In at least one embodiment, the ratio of the atomization gas to the high melting point metal in the atomization area may be about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize.

[00119] In at least one embodiment, the high melting point metal may be an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au. .

[00120] In at least one embodiment, the high melting point metal may be an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

[00121] In at least one embodiment, the high melting point metal is Cu.

[00122] In at least one embodiment, the high melting point metal is Sn.

[00123] In at least one embodiment, the process may comprise providing a high melting point alloy.

[00124] In at least one embodiment, the high melting point alloy may have a liquidus between about 500° Celsius to about 1800° Celsius.

[00125] In at least one embodiment, the high melting point alloy may have a liquidus of about 500° Celsius to about 150000° Celsius.

[00126] In at least one embodiment, a ratio of atomization gas to the high melting point alloy may be about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal.

[00127] In at least one embodiment, a ratio of atomization gas to the high melting point alloy may be about 5000 to about 40 000 cm^3 of gas per cm^3 of metal.

[00128] In at least one embodiment, the high meting alloy may comprise at least one element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

[00129] In at least one embodiment, the high meting point alloy comprises Cu and Sn.

[00130] In at least one embodiment, the high meting point alloy comprises Cu.

[00131] In at least one embodiment, the high meting point alloy comprises Sn.

[00132] In at least one embodiment, the high melting point allow consists essentially of Cu and Sn.

[00133] In at least one embodiment, the high melting point allow consists of Cu and Sn.

[00134] In at least one embodiment, the atomization gas stream may have a velocity of about 300 m/s to about 700 m/s. In at least one embodiment, the atomization gas stream may have a velocity of about 450 m/s to about 600 m/s. In at least one embodiment, the atomization gas stream may have a supersonic speed.

[00135] In at least one embodiment, the atomization gas may be delivered to an atomization head through at least one gas inlet 314, 311 oriented in a non-perpendicular

way with respect to the metal feed tube axis 312, providing a swirl movement of the atomization gas stream 240 in the atomization head 222 prior to the gas exit.

[00136] In at least one embodiment, at least two gas inlets 311, 314 may be tangential versus the central axis 312 of the feed tube 310. This configuration may create a dynamic rotational effect around the central axis 312 of the atomization plume in the atomization chamber 108.

[00137] In at least one embodiment, a distribution of powder particle sizes with geometric standard deviation may be lower than or about 2.2. In at least one embodiment, a distribution of powder particle sizes with geometric standard deviation may be of about 1.5 to about 2.2.

[00138] In at least one embodiment, a distribution of powder particle sizes with geometric standard deviation may be lower than or about 1.8. In at least one embodiment, a distribution of powder particle sizes with geometric standard deviation may be of about 1.5 to about 2.0.

[00139] In at least one embodiment, the atomization chamber 108 may comprise about 0 to about 20% of oxygen.

[00140] In at least one embodiment, the water may comprise at least one additive to control the redox potential of the water. Examples of additives comprise but are not limited to ethanol, methanol, acetic acid, HCl, H_2O_2.

[00141] In at least one embodiment, powder average particle size may be of about 10 microns to about 50 microns in diameter.

[00142] In at least one embodiment, the melt of the high melting point metal may be diverted through at least one melt diverting channel and the diverting angle is formed between the central axis of the feed tube and the at least one melt diverting channel.

[00143] In at least one embodiment, the alloy melt may be diverted through at least two melt diverting channels (diverters) 216 and the diverting angle (90°-Beta) may be formed between the central axis 212 of the feed tube 210 and the at least two melt diverting channels 216.

[00144] In at least one embodiment, at least one jet of water is sprayed into the atomization chamber 108.

[00145] In at least one embodiment, the at least one jet of water is sprayed on at least one wall of the atomization chamber 108.

[00146] In at least one embodiment, a powder may have an average particle size of less than about 50 microns. In at least one embodiment, a powder may have an average particle size of less than about 350 microns.

[00147] In at least one embodiment, the produced powder may be dried in vacuum to avoid powders oxidation.

[00148] In at least one embodiment, the produced powder may be washed with an organic solvent to remove most of the water prior of the drying stage. For example, the organic solvent may be ethanol or methanol.

[00149] In at least one embodiment, the atomization device 150 for manufacturing high melting point metal or alloy powder includes a feed tube 210 for providing a melt of said high melting point metal or alloy; a diverter 216, in fluid flow communication with said feed tube 210, for diverting the melt at a diverting angle with respect to a central axis of the feed tube 210 to obtain a diverted melt, and to directing the diverted melt to an atomization area 230 of the atomization device 150; at least one atomization gas injector 214 for providing at least one atomization gas stream 240 to the atomization area located inside the atomization chamber 108; and at least one water inlet 122 for providing water within an atomization chamber 108 of said atomization device 150.

[00150] In at least one embodiment, the diverter 216 may have a melt diverting conduit 218, the diverting conduit 218 being oriented at a diverting angle with respect to a central axis 212 of the feed tube 210.

[00151] In at least one embodiment, the diverter 216 may have at least two melt diverting conduits 218, each of the at least two melt diverting conduits 218 being oriented at a diverting angle with respect to a central axis 212 of the feed tube 210.

[00152] In at least one embodiment, the atomization device 150 may have at least one gas inlet 214 (or 311, 314). The at least one gas inlet 311, 314 of an exemplary

embodiment of the atomization device 300 may be tangential or at least non perpendicular to the atomization head 310 to provide a swirl movement of the atomization gas stream 240, in the atomization head 222 and a dynamic rotational movement of the atomization plume in the atomization chamber 108.

[00153] In at least one embodiment, at least one non perpendicular gas inlets (e.g. 311, 314) with respect to the atomization manifold 310 may create a swirl movement of the atomization gas stream 240 in the atomization head 222 leading to a dynamic rotational movement of the atomization plume in the atomization chamber 108.

[00154] In at least one embodiment, at least two gas inlets 214 may be non perpendicular to the atomization head 222 creating a swirling effect in the atomization head 222 and a dynamic rotational effect in the atomization area 230 and the atomization chamber 108.

[00155] In at least one embodiment, the at least one water inlet (e.g. 122 or 120 on Fig. 1) may be located inside the atomization chamber 108.

[00156] In at least one embodiment, the at least one water inlet (e.g. 122 or 120 on Fig. 1) may be suitable for providing water for cooling said powder.

[00157] For example, the at least one water inlet (e.g. 122 or 120 on Fig. 1) may be suitable for providing water for transporting said powder to the sieving/drying area.

[00158] In at least one embodiment, the at least one water inlet can be suitable for providing water for facilitating sorting/sieving of the powder.

[00159] **EXAMPLES**

[00160] **EXAMPLE 1: Copper atomized with different conditions**

[00161] In this test, the atomization of pure copper was carried out in a laboratory scale atomizer with a batch size of 3 Kg using the atomization manufacturing process and the atomization device as described herein. Three different conditions were tested to validate the effectiveness of the atomization device and the reproducibility.

[00162] Table 1A shows the atomization conditions used for the four tests of example 1.

Test no	Gas feed rate, g/sec	Averaged gas velocity, m/sec	Metal feed rate, kg/min	Gas to metal volume ratio
AG15-20	74	589	3.4	8518
AG15-22	110	635	1.7	25261
AG15-23	145	667	1.3	44455

Table 1A. Atomization conditions applied for three tests of example 1

[00163] The resulting average particle size and standard deviation are shown below. In all cases, sigma was below 2.0, which, in combination with the relatively low D50 obtained, led to very high percentage of particles between 1 to 50 µm. It is also clear that increasing the gas to metal volume ratio as well as the gas velocity led to a decrease of both D50 and sigma.

Test no	D50, µm	sigma	<50 µm, %	>50 µm, %
AG15-20	52	1.84	47	53
AG15-22	40	1.74	66	34
AG15-23	34	1.53	82	18

Table 1B Resulting averaged particle size and standard deviation.

[00164] **EXAMPLE 2: Copper**

[00165] In this exemplary test, the atomization of pure copper was carried out in a large atomizer with a batch size of 15 kg using the atomization manufacturing process and the atomization device as described herein.

[00166] Table 2A shows the atomization conditions of the test of Example 1.

Gas feed rate, g/sec	Averaged Gas velocity,	Metal feed rate, kg/min	Gas to metal volume ratio
132	560 m/s	1.5	34750

Table 2A. Atomization conditions applied in the test of Example 1.

[00167] The resulting average particle size and standard deviation are shown below. Considering the gas to metal volume ration and the average gas velocity used for this trial, the D50 and sigma are in quite good accordance with previous results obtained in a different atomizer.

D50, µm	Sigma
48	1.8

Table 2B. Resulting averaged particle size and standard deviation.

[00168] Figures 4A and 4B show SEM pictures of the powder obtained in the Example 2.

[00169] Morphology as determined with a Malvern Morphology equipment was measured. The circularity of the powder particles was about 0.992 in the 15-25 microns size fraction and 0.972 in particles size over 25 microns (the circularity is 1 for perfect spheres).

[00170] **EXAMPLE 3 : Copper Atomization**

[00171] In the tests of the Example 3 pure copper was atomized with two different atomizer to show the benefit of using the novel atomization technology compared to a conventional “converging-diverging” gas atomizer. Seven atomizations were realized with the new system and compared with +30 atomizations with the conventional technology. Results indicated a standard deviation in particle size significantly better that the conventional technology leading to much higher recoveries of powders in a prescribed size distribution range.

[00172] Figures 5A and 5B illustrate the benefit of the new atomization technology (new-C) compared to a reference conventional “Convergent-Divergent (conv-CD)” atomizer, wherein Figure 5A indicates a lower standard deviation in size distribution for the new technology and Figure 5B indicates a higher yield inside a prescribed particle size range for the new technology.

[00173] **EXAMPLE 4: Copper-Tin alloys**

[00174] Copper-Tin alloys were atomized using the atomization manufacturing process and the atomization device as described herein. Table 4A summarizes the conditions:

ID	Composition	Average gas velocity	Gas feed rate (g/s)	Metal feed rate (kg/min)	Gaz to metal volume ratio
AFA153	90% Cu – 10% Sn	568 m/s	125	2.5	19197
AFA173	75% Cu – 25% Sn	568 m/s	125	2.8	16352
AFA182	65% Cu – 35% Sn	568 m/s	125	2.8	16524

Table 4A. Atomization conditions applied in the three tests of Example 4.

[00175] The powders atomized using the above parameters in Table 4A display log-normal distributions with the fitting parameters described in Table 4B

ID	D50 (um)	Sigma
AFA153	24	1.7
AFA173	24	2.1
AFA182	19	2.2

Table 4B Resulting averaged particle size and standard deviation.

[00176] The embodiments of paragraphs [0012] to [00175] of the present disclosure are presented in such a manner in the present disclosure so as to demonstrate that every combination of embodiments, when applicable can be made. These embodiments have thus been presented in the description in a manner equivalent to making dependent claims for all the embodiments that depend upon any of the preceding claims (covering the previously presented embodiments), thereby demonstrating that they can be combined together in all possible manners. For example, all the possible combination, when applicable, between the embodiments of paragraphs [0012] to [00175] and the processes of paragraphs [0006] to [0011] are hereby covered by the present disclosure.

[00177] The scope of the claims should not be limited by specific embodiments and examples provided in the disclosure, but should be given the broadest interpretation consistent with the disclosure as a whole.

CLAIMS:

1. A high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt;
directing the diverted melt to an atomization area; and
providing at least one atomization gas stream to the atomization area,
said atomization process being carried out in the presence of water within an atomization chamber used for said atomization process.

2. A high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
delivering said melt through a diverter to an atomization area;
providing at least one atomization gas stream to the atomization area;
delivering water to an atomization chamber used for said atomization process, wherein, prior to being delivered to the atomization area, the melt is diverted in the diverter at a diverting angle with respect to a central axis of the feed tube.

3. A high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;
directing the melt to an atomization area; and providing at least one atomization gas stream having an average gas velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder with an average particle diameter under 50 microns with geometric standard deviation of lower than about 2.2.

4. A high melting point metal or alloy powder atomization manufacturing process comprising:

providing a melt of said high melting point metal or alloy through a feed tube;

optionally diverting said melt at a diverting angle with respect to a central axis of the feed tube to obtain an optionally diverted melt;

directing the optionally diverted melt to an atomization area; and

providing at least one atomization gas stream having a velocity of at least 300 m/s, to the atomization area, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000-cm^3 of gas per cm^3 of metal to atomize, thereby providing a distribution of powder particle sizes having geometric standard deviation of lower than about 2.2.

5. The process of any one of claims 1, 2 or 4, wherein the diverting angle (90-Beta) is about 30 to about 70 degrees.

6. The process of any one of claims 1, 2 or 4, wherein the diverting angle is about 10 to about 90 degrees.

7. The process of any one of claims 1 to 4, wherein an angle formed between the atomization gas and the melt is about 10 to about 90 degrees.

8. The process of any one of claims 1 to 4, wherein an angle formed between the atomization gas and the melt is about 40 to about 90 degrees.

9. The process of any one of claims 1 to 8, wherein the process comprises providing a high melting point metal.

10. The process of claim 9, wherein the high melting point metal has a melting point of about 500° Celsius to about 1800° Celsius.

11. The process of any one of claims 8 to 10, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal to atomize.

12. The process of any one of claims 8 to 10, wherein a ratio of the atomization gas to the high melting point metal in the atomization area is about 5 000 to about 40 000 cm^3 of gas per cm^3 of metal to atomize.

13. The process of any one of claims 1 to 12, wherein the high melting point metal is an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd and Au.

14. The process of any one of claims 1 to 12, wherein the high melting point metal is an element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

15. The process of any one of claims 1 to 12, wherein the high melting point metal is Cu.

16. The process of any one of claims 1 to 8, wherein the process comprises providing a high melting point alloy.

17. The process of claim 16, wherein the high melting point alloy has a liquidus of about 500° Celsius to about 1800° Celsius.

18. The process of claim 16, wherein the high melting point alloy has a liquidus of about 500° Celsius to about 1500° Celsius.

19. The process of any one of claims 13 to 18, wherein a ratio of atomization gas to the high melting point alloy is about 15 000 to about 30 000 cm^3 of gas per cm^3 of metal.

20. The process of any one of claims 13 to 18, wherein a ratio of atomization gas to the high melting point alloy is about 5000 to about 40 000 cm^3 of gas per cm^3 of metal.

21. The process of any one of claims 16 to 20, wherein the high melting alloy comprises at least one element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd and Au.

22. The process of any one of claims 16 to 20, wherein the high melting alloy comprises at least one element chosen from Al, Fe, Ni, Co, Cr, Mn, Si, Ti, Ag, Cu, Mo, Pt, Pd, Au and Sn.

23. The process of any one of claims 16 to 20, wherein the high melting alloy comprises Cu and Sn.

24. The process of any one of claims 16 to 20, wherein the high melting alloy comprises Cu.

25. The process of any one of claims 16 to 20, wherein the high melting alloy comprises Sn.

26. The process of any one of claims 16 to 20, wherein the high melting alloy consists essentially of Cu and Sn.

27. The process of any one of claims 16 to 20, wherein the high melting alloy consists of Cu and Sn.

28. The process of any one of claims 1 to 27, wherein the atomization gas stream has a velocity of about 300 m/s to about 700 m/s.

29. The process of any one of claims 1 to 27, wherein the atomization gas stream has a velocity of about 450 m/s to about 600 m/s.

30. The process of any one of claims 1 to 27, wherein the atomization gas stream has a supersonic speed.

31. The process of any one of claims 1 to 30, wherein the atomization gas is delivered to an atomization head through at least one gas inlet oriented in a non-perpendicular way with respect to the atomization head, the gas inlet providing a swirl movement in the atomization head prior to the gas exit.

32. The process of any one of claims 1 to 30, wherein at least two gas inlets to the atomization head are offset versus the central axis of the feed tube, creating a dynamic rotational effect around the central axis in the atomization area.

33. The process of any one of claims 1 to 32, thereby providing a distribution of powder particle sizes with geometric standard deviation of lower than or about 2.2.

34. The process of any one of claims 1 to 32, thereby providing a distribution of powder particle sizes with geometric standard deviation of about 1.5 to about 2.0.

35. The process of any one of claims 1 to 34, wherein the atomization chamber comprises about 0 to about 20% of oxygen.

36. The process of any one of claims 1 to 30, wherein the water comprises at least one additive to reduce the redox potential of the water.

37. The process of any one of claims 1 to 36, wherein the redox potential of the water has been reduced prior to the atomization.

38. The process of any one of claims 1 to 36, where the temperature of the water used in the atomization chamber is lowered so as to reduce the powders oxidation in the atomization process

39. The process of any one of claims 1 to 38, wherein the melt of said high melting point metal is diverted through at least one melt diverting channel and the diverting angle is formed between the central axis of the feed tube and the at least one melt diverting channel.

40. The process of any one of claims 1 to 38, wherein the alloy melt is diverted through at least two melt diverting channels and the diverting angle is formed between the central axis of the feed tube and the at least two melt diverting channels.

41. The process of any one of claims 1 to 40, wherein at least one jet of water is sprayed into the atomization chamber.

42. The process of any one of claim 41, wherein the at least one jet of water is sprayed on at least one wall of the atomization chamber.

43. The process of any one of claims 1 to 42, thereby providing a powder having an average particle size of less than about 50 µm.

44. The process of any one of claims 1 to 42, thereby providing a powder having an average particle size of less than about 35 µm.

45. The process of any one of claims 1 to 42, thereby providing a powder having an average particle size of about 10 µm to about 50 µm.

46. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 70 % of the powder has an average particle size of less than about 50 µm.

47. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 80 % of the powder has an average particle size of less than about 50 µm.

48. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 85 % of the powder has a particle size of less than about 50 µm.

49. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 70 % of the powder has a particle size of about 1 µm to about 40 µm.

50. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 75 % of the powder has an average particle size of about 1 µm to about 40 µm.

51. The process of any one of claims 1 to 42, thereby providing a powder in which at least about 80 % of the powder has an average particle size of about 1 µm to about 40 µm.

52. The process of any one of claims 1 to 51 in which the produced powder is vacuum dried to avoid powders oxidation.

53. The process of any one of claims 1 to 52 in which the produced powder is washed with an organic solvent to remove most of the water prior of the drying stage.

54. An atomization device for manufacturing high melting point metal or alloy powder, the device comprising:

a feed tube for providing a melt of said high melting point metal or alloy;

a diverter, in fluid flow communication with said feed tube, for diverting the melt at a diverting angle with respect to a central axis of the feed tube to obtain a diverted melt, and to directing the diverted melt to an atomization area of the atomization device;

at least one atomization gas injector for providing at least one atomization gas stream to the atomization area located inside the atomization chamber; and

at least one water inlet for providing water within an atomization chamber of said atomization device.

55. The atomization device of claim 54, wherein the diverter comprises a melt diverting conduit, the diverting conduit being oriented at a diverting angle with respect to a central axis of the feed tube.

56. The atomization device of claim 54, wherein the diverter comprises at least two melt diverting conduits, each of the at least two melt diverting conduits being oriented at a diverting angle with respect to a central axis of the feed tube.

57. The atomization device of any one of claims 54 to 56, further comprising at least one gas inlet, the at least one gas inlet being non perpendicular to the atomization head as to provide a swirl movement in the atomization head and a dynamic rotational movement in the atomization area and the atomization chamber.

58. The atomization device of any one of claims 54 to 56, wherein at least one non perpendicular gas inlets creates a circular flow in the atomization head leading to a dynamic rotational movement of the gas in the atomization area and the atomization chamber.

59. The atomization device of any one of claims 54 to 56, wherein at least two gas inlets are non-perpendicular to the atomization head creating a swirling effect in the atomization head and a dynamic rotational effect in the atomization area and the atomization chamber.

60. The atomization device of any one of claims 54 to 56, wherein the at least one water inlet is located inside the atomization chamber.

61. The atomization device of any one of claims 54 to 60, wherein the at least one water inlet is suitable for providing water for cooling said powder.

62. The atomization device of any one of claims 54 to 60, wherein the at least one water inlet is suitable for providing water for transporting said powder to the sieving/drying area.

63. The atomization device of any one of claims 54 to 60, wherein the at least one water inlet is suitable for providing water for facilitating sorting/sieving of said powder.

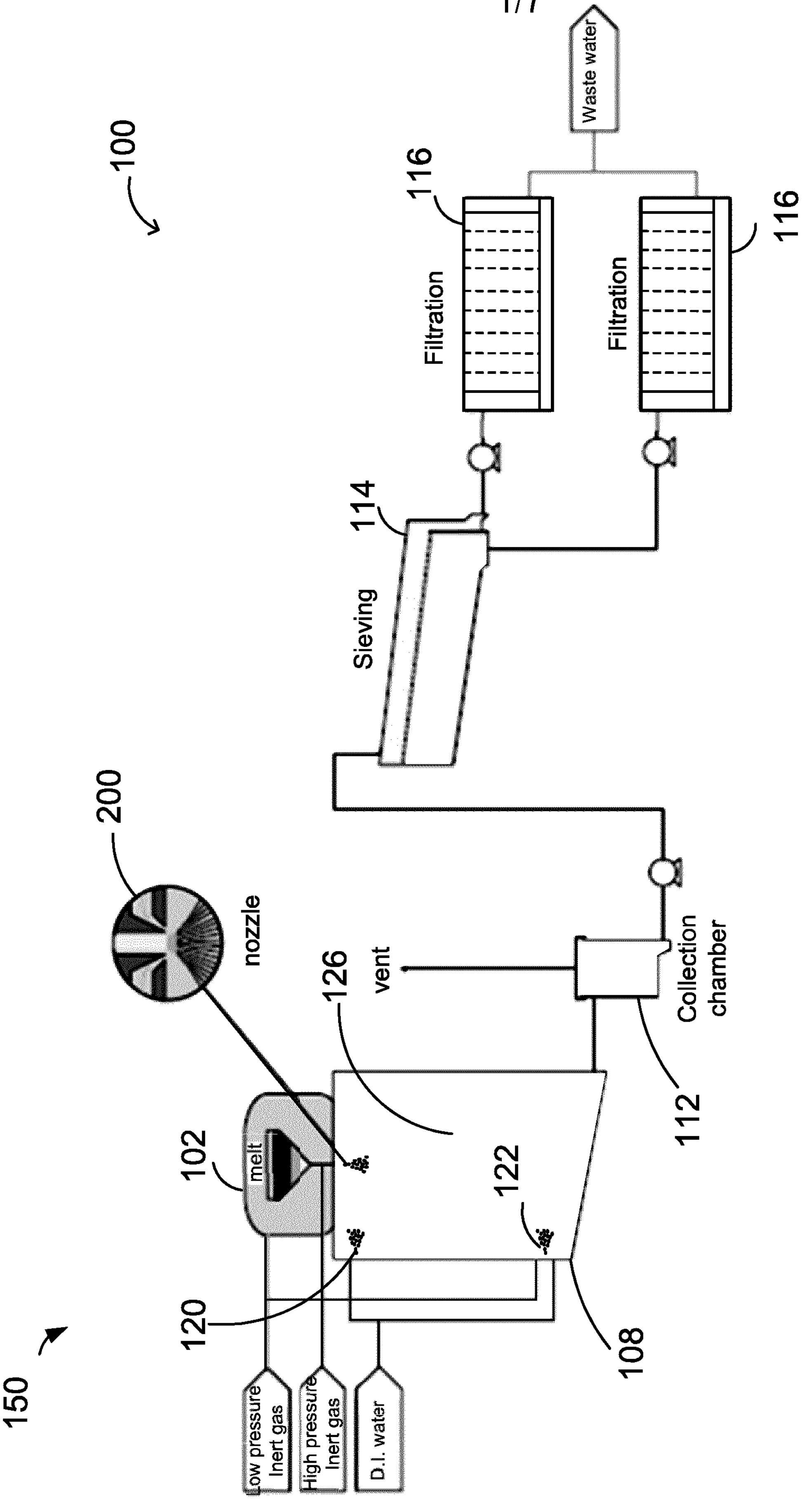
100
150
Waste water
116
116
Filtration
Filtration
114
Sieving
200
nozzle
126
vent
Collection chamber
112
102
melt
122
120
108
Low pressure Inert gas
High pressure Inert gas
D.I. water

FIG. 1

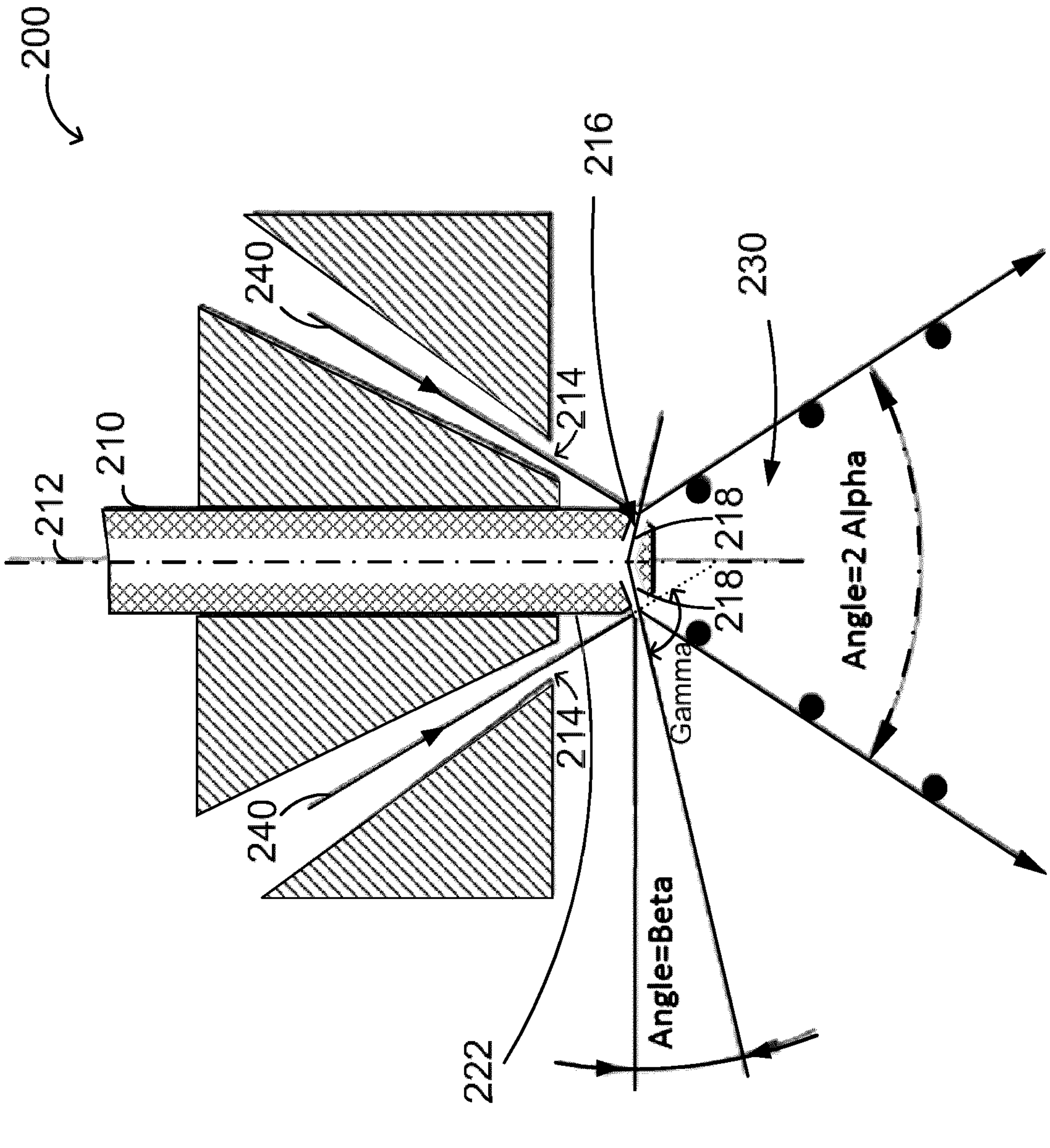
200
212
210
216
240
214
230
218
218
Angle=2 Alpha
Gamma
214
240
Angle=Beta
222

FIG. 2

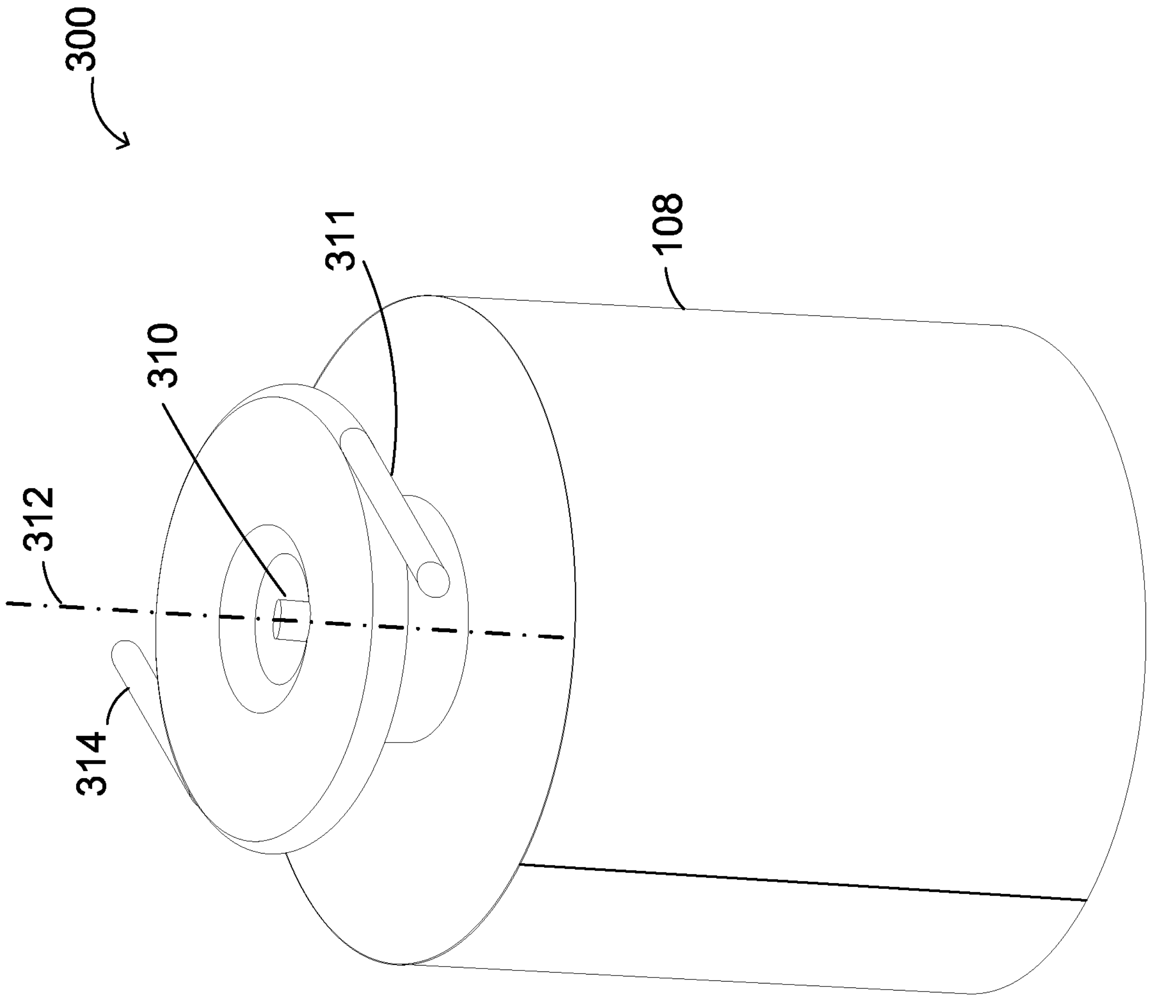

FIG. 3

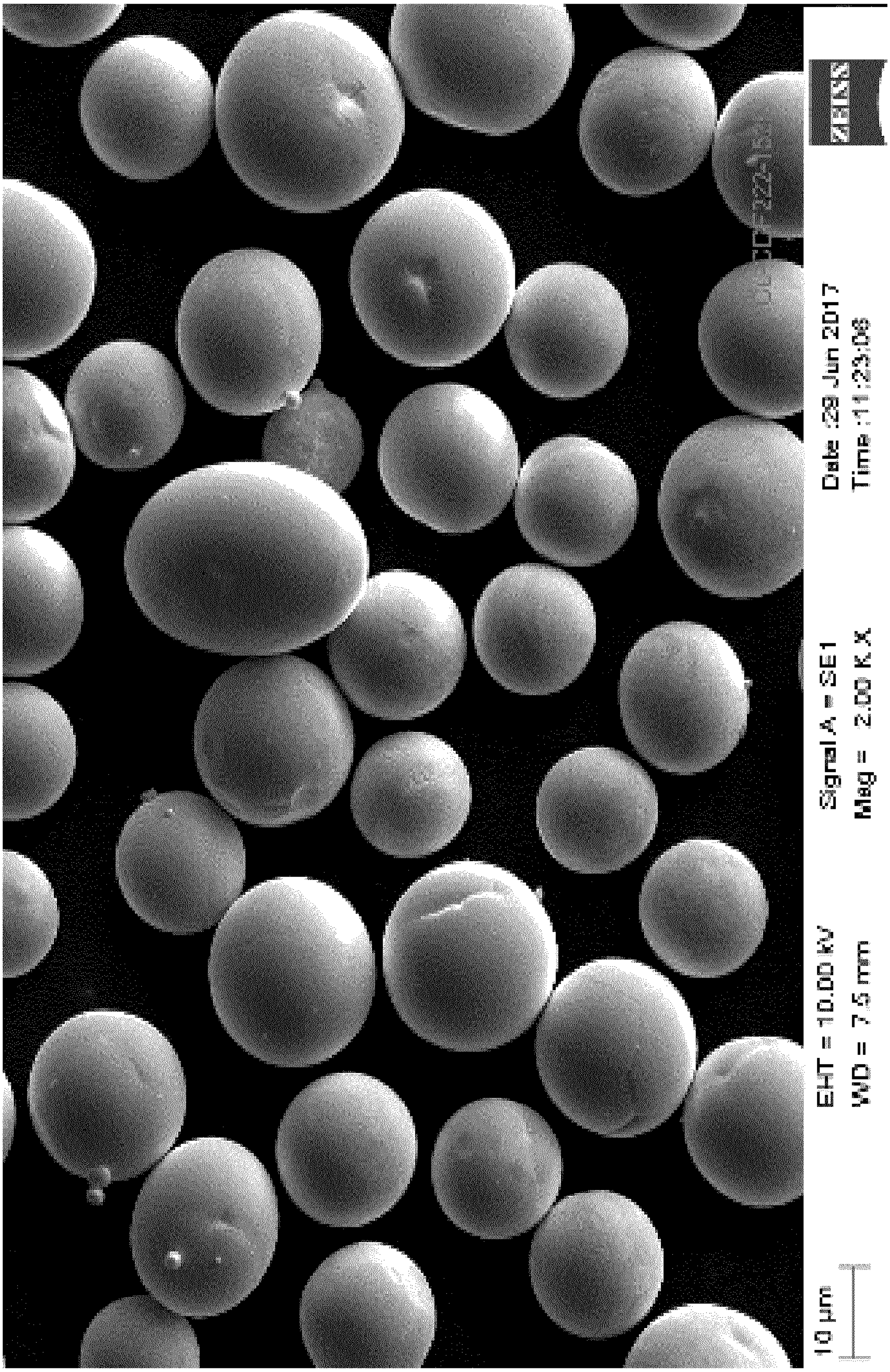
ZEISS
EHT = 10.00 kV
WD = 7.5 mm
Signal A = SE1
Mag = 2.00 K X
Date :29 Jun 2017
Time :11:23:08
10 µm

FIG. 4A

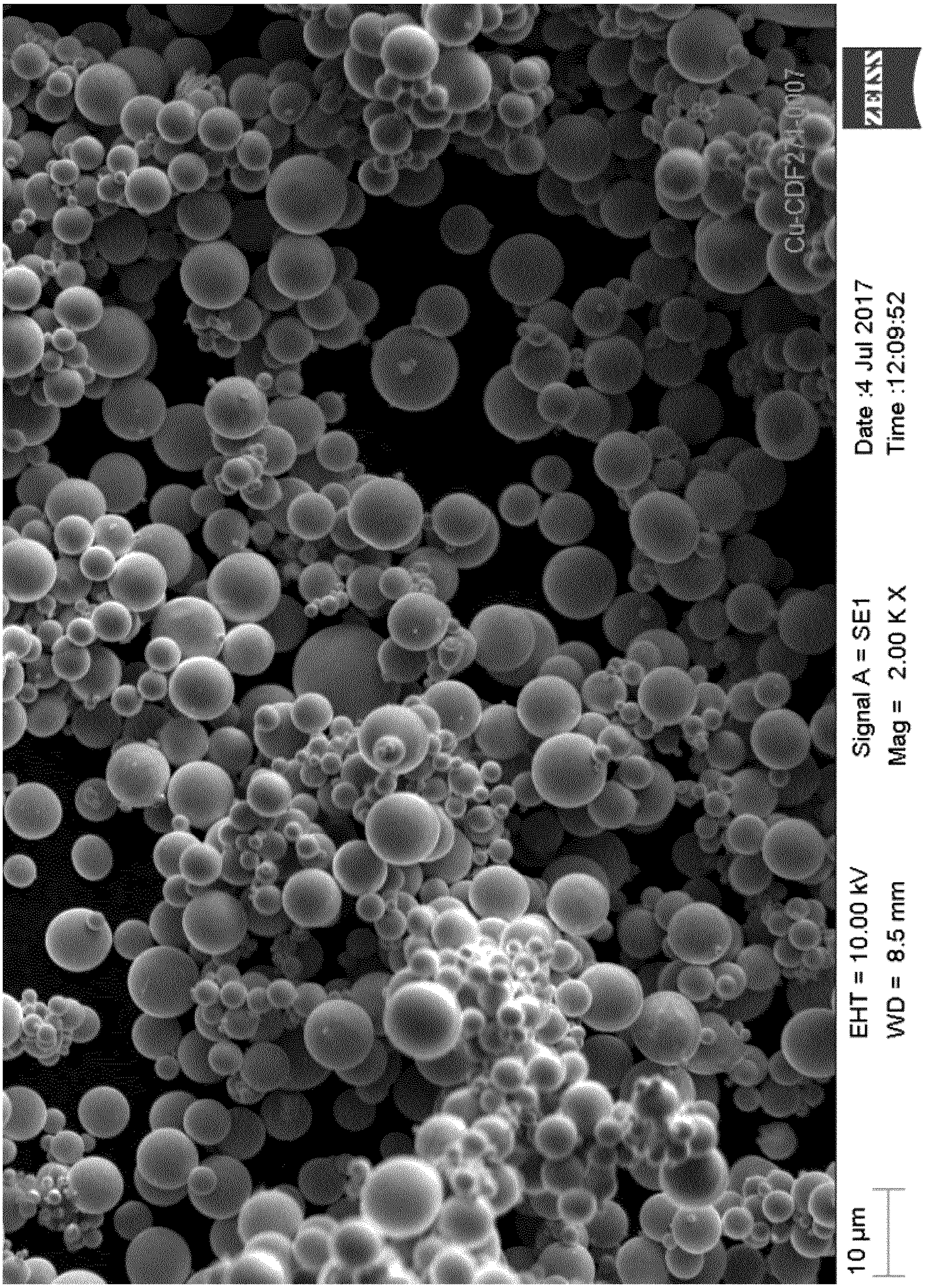
10 µm
EHT = 10.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 2.00 K X
Date :4 Jul 2017
Time :12:09:52

FIG. 4B

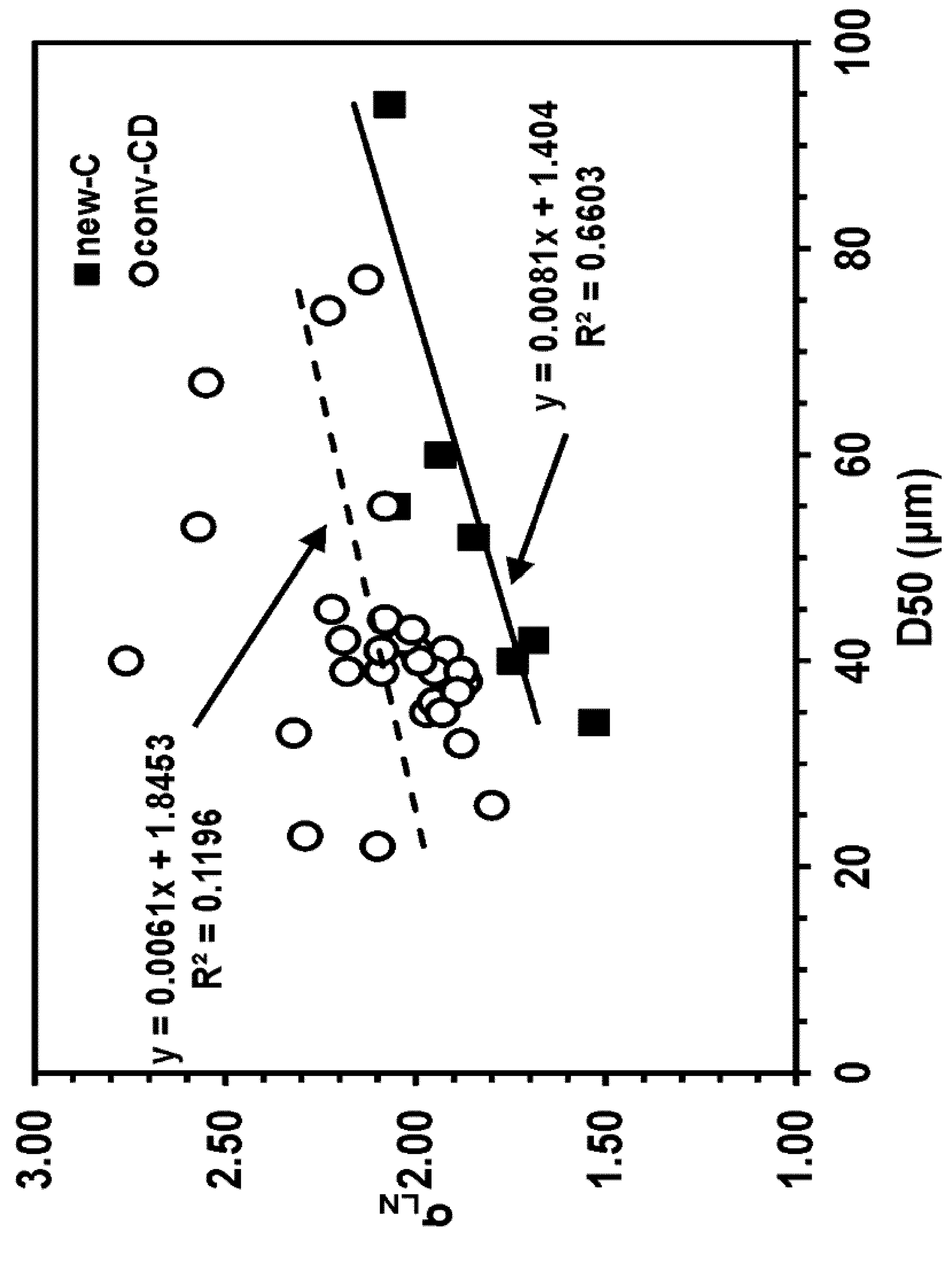
■new-C
Oconv-CD
y = 0.0081x + 1.404
R² = 0.6603
y = 0.0061x + 1.8453
R² = 0.1196
D50 (µm)
0
20
40
60
80
100
3.00
2.50
2.00
1.50
1.00

FIG. 5A

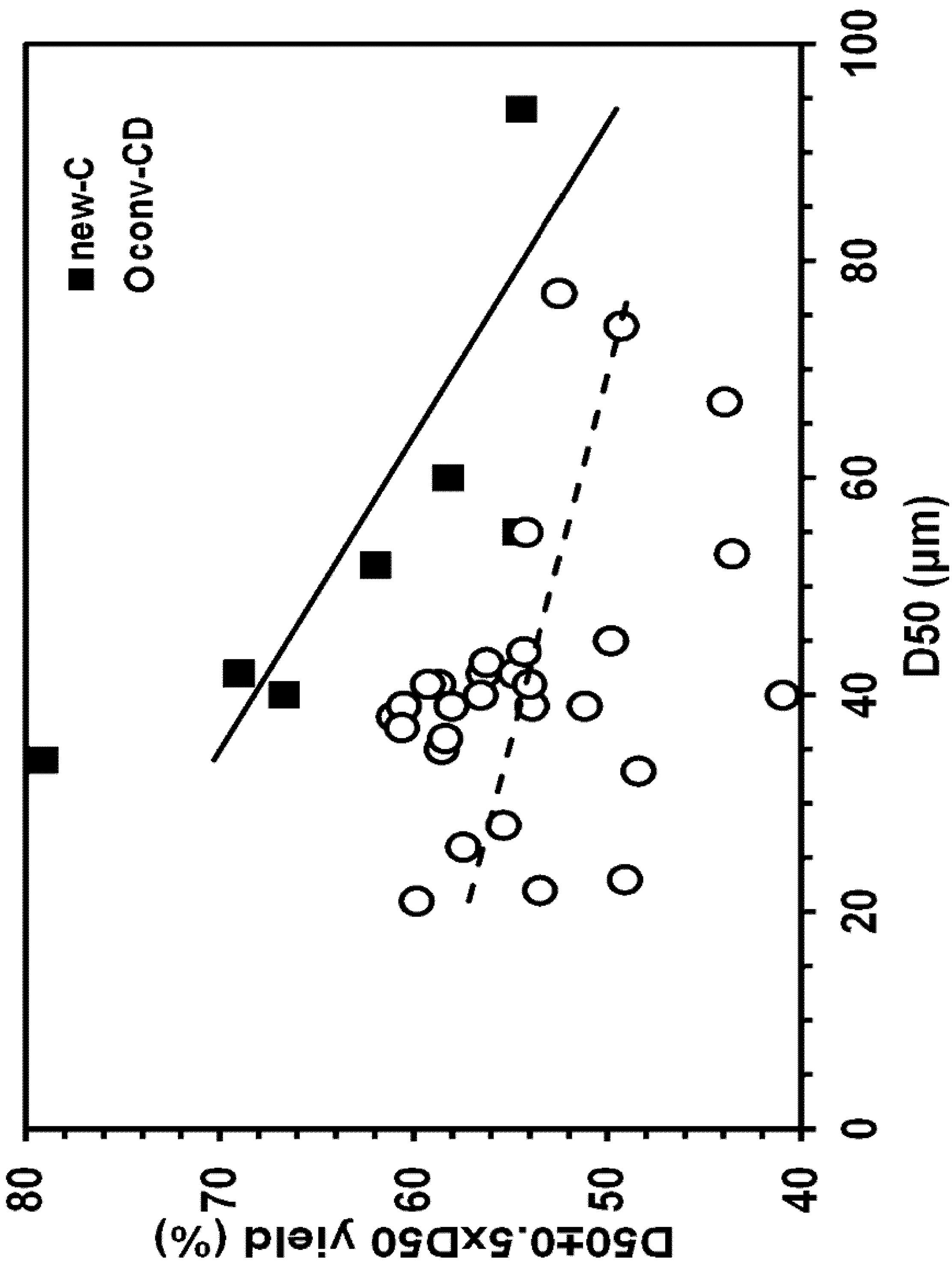
new-C
conv-CD
D50 (µm)
D50±0.5xD50 yield (%)
0
20
40
60
80
100
40
50
60
70
80

FIG. 5B

CORRECTED VERSION

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2019/050176

A. CLASSIFICATION OF SUBJECT MATTER
IPC: ***B22F 9/08*** (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC: B22F*

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)

Keywords searched:
1. ((metal or alloy) 1W powder) and atomi* gas and (molten or melt*) and (tube or pipe) and supersonic and (swirl* or rotat*))
2. (atomi* gas and (reactor or chamber) and water and (cool* or quench*) and powder and (redox potential or ethanol or methanol or formic acid or acetic acid) and oxidation)
3. (atomiz* gas and ((metal or alloy) 1W powder) and (molten or melt*) and supersonic and (ratio S gas S (metal or alloy))

Publication Date: =< 12 February 2019 (12-02-2019); IPC = B22F*

Databases searched and search string:
Search #1: Keywords (1) + Publication Date; Search #2: Keywords (2) + Pub. Date; and Search #3: Keywords (3) + Pub. Date

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US 2016/0023277 A1 (RIEKEN, J.R. et al.) 28 January 2016 (28-01-2016) Paragraphs 0003, 0009, 0024- 0026 & 0056; claims 1, 12 & 15; and Figs. 1a & 3b.	9-30, 33-35 & 43-53 1-8, 31, 32, 36-42 & 54-63
Y	US 6,142,382 (TING, J. et al.) 7 November 2000 (07-11-2000) Abstract; col. 2, lines 13-23 & 34-64; col. 9, lines18-56; claims 1, 4 & 7; and Figs. 1E, 12 & 16.	5-8, 39, 40, 55 & 56
Y	US 2017/0144227 A1 (NAKASEKO, M. et al.) 25 May 2017 (25-05-2017) Abstract; paragraphs 0001, 0006, 0008, 0018, 0020, 0073, 0076, 0084-0086, 0090 & 0091; claims 5 & 8; and Fig. 3.	1, 2, 36-38, 41, 42, 54 & 60-63

☑ Further documents are listed in the continuation of Box C.
☑ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search
8 April 2019 (08-04-2019)

Date of mailing of the international search report
01 May 2019 (01-05-2019)

Name and mailing address of the ISA/CA
Canadian Intellectual Property Office
Place du Portage I, C114 - 1st Floor, Box PCT
50 Victoria Street
Gatineau, Quebec K1A 0C9
Facsimile No.: 819-953-2476

Authorized officer
Benjamin Chan (819) 639-8445

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CA 1,315,055 C (FISCHER, J.J.) 30 March 1993 (30-03-1993) Abstract; page 1, lines 6-9; page 2, lines 5-14; page 3a, lines 8-11& 18-20; page 4, lines 8-12 & 17-19; page 7, lines 24-37; and claims 1, 5 & 6.	1, 2, 36-38, 41, 42, 54 & 60-63
Y	US 4,272,463 (CLARK, I.S.R. et al.) 9 June 1981 (09-06-1981) Abstract; col. 1, lines 6-9; col. 2, lines 3-6; col. 7, lines 50-67; and claim 1.	3, 4, 31, 32 & 57-59
Y	US 4,988,464 (RILEY, M.F.) 29 January 1991 (29-01-1991) Abstract; col. 1, lines 6-53; col. 2, line 56 to col.3, line 16; col. 3, lines 49-68; col. 4, lines 22-23, 27-29 & 47-61; and claims 1, 4 & 5.	3 & 4
Y	US 5,480,470 (MILLER, S.A. et al.) 2 January 1996 (02-01-1996) Abstract; col. 1, lines 25-30 & 50-54; col. 2, lines 65-67; col. 3, lines 1-23; col.4, lines 27-29; col. 6, lines 14-34; col. 9, lines 26-50; col. 13, lines 20-31; and claims 10 & 21.	3 & 4
Y	JP 2002-105514 A (OBIKA, M. et al.) 10 April 2002 (10-04-2002) Whole document (English machine Translation).	3 & 4
P, X, Y	CA 2,999,242 C (ST-LAURENT, S. et al.) 01 March 2018 (01-03-2018)	1-63

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of the first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claim Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

The claims are directed to a plurality of inventive concepts as follows:

Group A - claims 1, 2, 5-63 are directed to a gas atomization process and a gas atomization device for manufacturing high melting point metal or alloy powder. In the process, a melt of the high melting point metal or alloy is fed through a feed tube and diverted at a diverting angle with respect to a central axis of the feed tube to an atomization area, and water is provided within an atomization chamber of the device; and

Group B - claims 3-53 are directed to a gas atomization process for manufacturing metal or alloy powder. In the process, a melt of high melting point metal or alloy is fed through a feed tube into an atomization area. At least one atomization gas stream having a velocity of at least 300 m/s and a volume ratio of atomization gas to metal to be atomized of 5,000 to 40,000 is provided to the atomization area.

The common technical feature of Group A and Group B appears to be the application of a feed tube to feed a melt of high meting point metal or alloy to an atomization area. However, this feature is well-known in the art of gas atomization. Therefore, Group A and Group B do not have any other common technical features.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☑ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claim Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

Patent Document Cited in Search Report	Publication Date	Patent Family Member(s)	Publication Date
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US2017144227A1	25 May 2017 (25-05-2017)	US2017144227A1	25 May 2017 (25-05-2017)
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		CN106132599B	20 March 2018 (20-03-2018)
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		WO2018035599A1	01 March 2018 (01-03-2018)