APPARATUS FOR PRODUCING LOW OXIDE METAL POWDERS

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ABSTRACT

Apparatus is disclosed for producing high purity metal powders having an irregular and angular shape and a very low oxygen content (less than 0.25 percent oxygen in iron and steel powders). The invention utilizes a high pressure liquid atomization procedure for converting the molten metal to angular particulate form, and provides for the very rapid subsequent cooling of the hot particles under conditions of high pressure sprays and violent turbulence of the powder particles in the liquid that minimize the formation of oxide impurities on the particle surface. High pressure atomization to produce angular and irregular particles tends to create an oxidizing environment because of the mixture of hot particles and liquid. By rapidly quenching the particles immediately after formation, in a quenching environment that creates a violently turbulent condition at the surface of the metal, the formation of vapor or steam films is minimized and more rapid heat transfer from the particles to the cooling medium is realized.

16 Claims, 6 Drawing Figures
APPARATUS FOR PRODUCING LOW OXIDE METAL POWDERS

RELATED U.S. APPLICATION

This Application is a continuation-in-part of application Ser. No. 855,096, now U.S. Pat. No. 3,646,176, filed Sept. 4, 1969.

BACKGROUND OF THE INVENTION

Metal powders have gained increasing popularity in recent years mainly because of new, practical and commercially feasible methods for producing them. Metal powders can be produced by a number of processes including atomization of the molten metal by liquids or gases under pressure. A particularly advantageous method for the liquid atomization of molten metals, particularly iron or steel, is disclosed in my U.S. Pat. No. 3,334,408. Briefly, the method disclosed in the foregoing patent involves the use of high velocity, thin, solid, flat streams of cooling liquid that angularly impinge upon a stream of the molten metal to disperse it into fine, irregularly shaped powder particles. The powder particles thus formed are quenched and may be subsequently molded or compacted into coherent forms having many commercial applications.

I have found that the techniques adopted for the production of optimum powder shapes (i.e., irregular, angular) are inherently conducive to rapid surface oxide formation. Thus, iron powders produced by the liquid atomization of molten iron or steel generally have an oxygen content of more than about 0.7 percent after quenching and between about 0.8 percent and 1.0 percent after being dried. In order to use such iron powders for high quality products, (i.e., those requiring a low oxide impurity grade iron), the oxygen content of the powder should be reduced to less than about 0.25 percent. The removal of such oxide impurities from iron powders can be accomplished by annealing the powder in a reducing atmosphere in accordance with well-known procedures. However, the annealing process can have adverse effects on the powder, as by undesirably increasing the grain size. It also has been found that the annealing of iron powder relieves energy internal stresses in the particles which I have found to be advantageous for the subsequent processing of wrought products.

The oxidation of iron powder particles produced by liquid atomization of the molten metal is a function of many variables, including the particle size, time at elevated temperature, and environment. Iron powder will oxidize very rapidly at temperatures down to about 300°F. in an oxidizing environment. However, when cooled to below about 200°F., the oxidation rate is relatively slow. Oxide formation also, of course, occurs during the drying of liquid atomized powder, which tends to compound the problem of high oxide formation.

Hence, where low oxide powders have been required, it has been conventional to utilize gas atomizing techniques, rather than liquid atomization, to derive the metal powders. However, gas atomizing techniques have many significant disadvantages. For one thing, the production capacity of a gas atomizing system is very low, as there is a relatively low rate of heat transfer between the hot metal and the atomizing gas. Additionally, the cost of the atomizing gas, which must be inert, is a significant factor in the economics of the system.

Moreover, since the metal is cooled down at a relatively slow rate by gas atomization procedures, the atomized metal forms into particles of spherical shape, and particles of spherical shape are disadvantageous, as compared to irregular, angular particles produced by liquid atomization, for many end uses. Thus gas atomization has not provided a satisfactory answer to the production of low oxide atomized powder.

SUMMARY OF THE INVENTION

The present invention is directed to apparatus enabling low oxide atomized metal powders to be produced by liquid (typically water) atomizing procedures. This enables the high production capacities and favorable economics of the liquid atomizing techniques to be realized, and also accommodates the production of angularly shaped, irregular metal particles, which are advantageous for subsequent processing.

In accordance with the present invention, molten metal is subjected to liquid atomization in a procedure of two or more distinct but closely timed stages. In the first stage, a controlled stream of the molten metal is acted on by thin, flat, solid sheets of atomizing liquid, which are disposed to intersect in the form of a Vee and are ejected under extremely high pressure (e.g., about 500 psi or greater). The interception of the molten metal by the high pressure flat streams causes the molten metal stream to be shattered and dispersed into fine metal particles of the desired angular, irregular shape. Almost immediately thereafter, the atomized metal particles, still at high temperature, are struck by at least one and in some cases two or more additional sets of liquid jets, the function of which is to effect extremely rapid transfer of heat from the hot metal particles to the liquid by intimate contact between the water and the particles under conditions of substantial velocity and agitation. The hot particles are maintained continuously in highly turbulent contact with cooling liquid, until the particles are reduced to a temperature of, say, 200°F., at which temperature the tendency to oxidize is significantly reduced. Typically, the process is carried out by directing the particles, immediately after being struck by the subsequent stages of liquid jets, into a highly turbulent water body which disperses the particles and continues the cooling to a desired final level of around 200°F. or below.

In the process of the invention, an inert environment advantageously is maintained at the stages in which the metal is initially atomized and then quenched by jets of atomizing and cooling liquid, in order to reduce to a minimum the exposure of the metal to oxygen during its critical, higher temperature stages, to reduce the possibility of explosion on initial contact and to control the vacuum produced in the water leg formed by the turbulent water, metal particle flow after initial atomization and quenching. Obviously, this precludes any undue entry of air into the atomizing zone. Nevertheless, some oxygen will be present for reaction with the high temperature metal particles (e.g., from dissociation of the cooling water itself or from water vapor), and the rapid quenching of the atomized particles to a temperature below that at which oxidation reactions readily occur is a critical aspect of the present process. In this respect, once the molten metal stream is disintegrated into fine metal particles, the surface area available for oxidation reactions is enormously increased,
and the tendency to form oxides is correspondingly increased.

In accordance with a specific aspect of the invention, the atomization-quenching sequence is required to be carried out in two or more distinct stages, in order to achieve the combined results of a small, angular, irregularly shaped particle and a sufficiently low overall oxygen content. In this respect, in order to achieve the small, angular, irregular particle which is desired in accordance with the invention, it is necessary to intercept a descending metal stream, typically of 1/4 to 1/2 inch in diameter, with intersecting thin, flat, solid streams of liquid, typically water, at high pressure. If these atomizing streams are sufficiently thin (i.e., using spray nozzle openings about 1/32 to 1/16 of an inch in thickness) to achieve the desired particle size and shape, they lack sufficient liquid volume to achieve sufficient heat transfer from the particles in the region of water and hot metal contact to avoid substantial oxidation. In other words, I have found that the requirement of achieving a sufficiently high rate of heat transfer, on the one hand, and a desired particle shape and size, on the other, with a single set of liquid streams, are mutually inconsistent. Under the procedure of the invention, the atomized metal, at the moment immediately following atomization, is again forcibly struck by at least one additional set of liquid streams. The additional set or sets of streams are of sufficient thickness and volume to effect a high rate of heat transfer from the high temperature particles, and to rapidly cool the particles. At the second stage of liquid jets, the metal already has been substantially atomized, so that the additional stages of jets are controlled for optimum heat transfer.

After the quenching stage of jets, the particles are directed immediately into a turbulent body of cooling water which further cools the particles down to below 200°F. A condition of violent turbulence between the particles and quenching water must be maintained, until the particles are in a temperature below the boiling point of the water. This avoids any sustained contact of the metal surfaces with steam or water vapor, which are highly reactive, oxide-forming media.

Iron powder, for example, can be produced in accordance with the invention to have an oxygen impurity content at the extraordinarily low level of significantly less than 0.25 percent, even after drying, it being understood that, under normal circumstances, iron powder will be subject to some oxide formation (and therefore additional oxygen pick-up) during a drying step, because of the elevated temperature conditions necessarily involved in the economical drying of water atomized powders.

The techniques of the present invention are especially significant in the production of atomized powders from certain classes of metals and alloys. Many alloyed materials contain oxygen-reactive components such as chromium, aluminum, titanium, manganese, silicon, etc. The oxides formed with many of these reactive materials are difficult, if not impossible, to reduce in subsequent operations. Therefore, the techniques of water atomizing these materials under circumstances which substantially minimize the formation of oxides in the first place are especially valuable. Even in the case of ordinary iron and steel materials, where objectionable oxides formed during more conventional water atomizing processes might be reduced in subsequent stages, complications may arise quite apart from the unfavorable economics of the additional reducing operations, in the sense that the reducing operations typically are accompanied by grain growth. Thus, in the end, it may not be possible to achieve with conventional procedures the finer grain structures which are desirable for many end uses.

For a more complete understanding of the invention, reference should be made to the following detailed description and to the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a simplified, schematic representation of a liquid atomization apparatus incorporating the principles of the invention;

FIG. 2 is an enlarged cross section taken generally on line 2—2 of FIG. 1;

FIG. 3 is an enlarged cross section taken generally on line 3—3 of FIG. 2;

FIG. 4 is a modification of the apparatus of FIG. 1, showing a further embodiment of the principles of the invention;

FIG. 5 is an enlarged cross section taken generally on line 5—5 of FIG. 4; and

FIG. 6 is an enlarged cross section taken generally on line 6—6 of FIG. 5.

DESCRIPTION OF PREFERRED EMBODIMENTS

Referring now to the drawings in which like reference characters refer to like parts throughout the several views thereof, the reference numeral 10 in FIG. 1 designates a large open top receiving tank. The receiving tank retains a body of cooling liquid, typically water, designated by the reference numeral 11. The tank is also provided with an outlet opening 12 for removal of water and particulate matter, as will be described.

Suitably mounted on the receiving tank 10 is an atomizer housing 13. In accordance with the invention, the housing 13 constitutes a sealed enclosure. It is provided, however, with an opening 14 at the top for the introduction of molten metal for atomization, with an opening 15 in an upper portion thereof for admitting inert gas, and with a discharge opening 16 in its lower extremity, below the water level in the retaining tank 10. As indicated in FIG. 1, the discharge end 16 of the atomizer housing is of smaller dimensions than the upper portions thereof constituting the atomizing chamber 17. By way of example, in an apparatus of typical proportions for pilot-scale operations, the atomizing chamber portion of the housing may be of generally rectangular cross section, having a width dimension on the order of 15 inches and a thickness dimension on the order of 10 inches. The discharge opening 16, on the other hand, may have a thickness dimension (vertical in FIG. 1) on the order of 2–3 inches, with a width dimension on the order of 6 inches. Between the atomizing chamber 17 and the discharge opening 16 the atomizer housing advantageously tapers gradually in its thickness dimension and angles forwardly somewhat in a water leg section 18.

At the top of the atomizer housing 13 is a receiving crucible 19 adapted, when the system is in operation, to receive a body of molten metal 20. The crucible 19 has an opening 21 in its bottom wall, which communicates with the interior of the atomizer housing 13 and provides for the gravity discharge of molten metal in a solid stream. The descending stream of molten metal is on the order of 3/8 of an inch in diameter, although
larger sizes (e.g., 7/16 inch) may be utilized, as will be discussed below, in some instances.

In accordance with one embodiment of the invention, two sets of liquid spray jets are provided in the atomizing chamber 17, disposed to act in rapid sequence upon the descending stream of molten metal. As shown in FIG. 2, a first pair of water discharge nozzles 22, 23 is disposed symmetrically on opposite sides of the descending, coherent stream of molten metal 24. The nozzles 22, 23 are directed downward and inward at an angle of 15°-30° from the vertical, and are arranged to direct controlled jets of atomizing water into intercepting relation to the molten metal stream 24.

As a significant facet of the invention, it is important to derive, in the metal atomizing stage, metal particles which are fine in size and are angular and irregular in configuration. This is achieved through the use of thin, flat, solid streams 25, 26 of atomizing water, ejected from the nozzles 22, 23, at high pressure. For optimum results, the atomizing streams 25, 26 are ejected under pressures of 500-1,000 psi, typical for atomizing iron and steel in a pilot scale unit. Higher pressure may be desired for larger installations; lower pressure may be desired for certain, easily atomized metal. The nozzle openings, through which the water streams are ejected optimally are of rectangular configuration, measuring about 1/2 inch in width and 1/32 inch in thickness. These nozzles eject solid, flat streams 25, 26 of water from points around 5 inches or so away from the point 27 of intersection with each other and with the descending metal stream 24. In the short distance between the nozzle tip and the point of intersection 27, the water streams will fan out somewhat to a width of about 3 inches, as indicated in FIG. 2, and may increase slightly in thickness, but will retain their flat, thin, solid characteristic.

The interaction of the high pressure water streams 25, 26 with the descending molten stream 24 causes the molten metal stream to be literally shattered and broken up into fine particles. The particles are almost instantly solidified and, due to the violence and rapidity of the solidification, the particles are derived in an irregular and angular configuration which is highly desirable. In accordance with the invention, the interaction of the water streams 25, 26 and the descending stream of molten metal 24 is such as to produce particles substantially all of which are minus 40 mesh in size and preferably minus 80 mesh. This means that almost all of the particles produced would pass through a mesh screen within the range of between about 40-80 mesh (U.S. Sieve Series, A.S.T.M. specification E-11-61).

A critical facet of the present invention involves, in addition to the production of fine, atomized particles as described immediately above, the maintenance during the atomizing process under non-oxidizing conditions and, in addition, the quenching of the atomized particles in the earliest possible time to a temperature below which oxidation readily occurs. Extremely rapid quenching of the atomized particles is enabled, in part, by the production in the first instance of particles of suitable fineness, and so the conduct of the atomizing stage itself is an integral part of the invention. It has been observed, however, that the formation of atomized particles of the desired size and shape, and the sufficiently rapid transfer of heat from these particles tend to be mutually inconsistent objectives, at least when using a single set of nozzles. Accordingly, as an important part of the invention, at least one set of water nozzles 28, 29 is provided in the atomizing chamber, arranged to direct streams of water 30, 31 into intersecting impingement at 32, just slightly below the intersecting impingement 27 of the principal atomizing streams 25, 26. Advantageously, the water streams 30, 31 are brought as close up to the streams 25, 26 as practicable without causing interference with the action of those streams. In practice, in an atomizing apparatus of the general dimensions and configurations described, the second stage streams 30, 31 may intersect at a point from as close as about 1/4 inch to as far as about 2 inches below the intersection of the first stage streams, with a more typical spacing being about 3/4 inch.

The nozzles 28, 29 may be operated at a somewhat lower pressure than the first stage streams, say, on the order of 100 psi or more and typically around 350-400 psi, and may advantageously deliver water in solid streams of somewhat greater thickness than the atomizing streams, substantially as illustrated in FIG. 3. The objective in the case of streams 30, 31, is to envelope the just-atomized particles in a substantial volume of water, accompanied by violent turbulence. This provides for the fastest possible transfer of heat from the small metal particles to the quenching water, by minimizing sustained contact between the hot particles and unagitated water. This minimizes the formation of surface films of steam or vapor that tend to form during the quenching. In this respect, it will be understood, that steam is a highly reactive oxidizing medium, and surface oxides will quickly form and heat transfer will be impeded if there is sustained exposure of the particles to such steam films.

Most advantageously, even after exposure of the metal particles to the streams 30, 31, it is desirable to follow immediately with a further cooling stage, in order to bring the particles well below the temperature at which oxidation is promoted. In one practical form of the invention, the quenched particles are flowed downward through the water leg 18 of the atomizer housing, and ejected out through discharge opening 16 into the body of water 11. Desirably, the water issuing from the discharge nozzle 16 has sufficient forward discharge velocity to maintain substantial turbulence within the water body 11. However, supplementary agitation of the main body 11 of cooling water may be provided, if necessary or desirable. Advantageously, the water utilized for quenching and cooling may be heated to reduce its content of dissolved oxygen, to further reduce the exposure of the metal to oxidizing conditions.

In the practice of the invention, the range of particle sizes plays an important part, because there is a significant, inverse ratio between the mass of the individual particles and the surface area available for cooling contact (and also oxidation). In general, the smaller the particle size the better, up to a point. Heat is more readily extracted from a small particle, because of its favorable surface area-to-mass ratio. On the other hand, if the particles are too small, an excessive area is presented for possible oxidizing reaction, not only during quenching and cooling, but during subsequent drying, handling and storage. Moreover, if the particles are too small, compaction of the powder to form wrought products is made difficult. Optimum results in the practice of the invention are realized when the particles are within the range of between about minus 40 mesh...
minus 80 mesh, and preferably minus 80 mesh; advantageously, however, not more than about 60 percent of the particles are minus 325 mesh in size.

Notwithstanding the introduction of substantial quantities of water (e.g., at least 50 gallons per minute and in some instances 70–90 gallons in the pilot-sized equipment described) through the atomizing and quenching nozzles, and the introduction of metal, the action of the high velocity water jets within the atomizing chamber causes a substantial reduction in the ambient pressure of the chamber. This causes water to be drawn into the water leg 18 from the main cooling body 11, somewhat in the manner illustrated in FIG. 1. In a unit of the proportions described, a pressure reduction of 6 feet of water or more can occur, which means that the water would rise that far in the water leg 18 above the level of the water 11 in the absence of proper controls. Since the water retained internally of the atomizer housing 13 is in a state of violent turbulence, interminably and at the surface, steps must be taken to avoid interference of the drawn-up water with the action of the atomizing and quenching jets. Under certain conditions, it might be possible to accomplish this by simply increasing the height of the water leg 18, such that the atomizing nozzles were located sufficiently high above the level of the cooling water to avoid any interference. However, it appears that, when this is done, the just-quenched particles are retained too long in the warmer water of the water leg before reaching the body of cooling water 11. Accordingly, as an advantageous feature of the embodiment of the invention in FIG. 1, provisions are made for controllably increasing the pressure within the atomizing chamber 17, partially to compensate for the pressure reducing action of the atomizing and quenching jets, and thereby controlling the height of water in the water leg 18 and expediting passage of the atomized particles out through the discharge opening 16 and into the large body of cooling water.

In the system of FIG. 1, pressure in the atomizing chamber is controllably increased by means of a supply (not shown) of inert gas, typically argon, which is fed in through a conduit 33, through a flow or pressure regulator 34. In a typical operation of the described apparatus, the regulator pressure may be adjusted to achieve a desired level of water in the water leg 18, which, even allowing for substantial surface turbulence, will usually provide sufficient clearance below the atomizing and quenching jets to avoid interference. The out-gassing of the metal itself may be utilized to advantage in controllably increasing the pressure in the atomizing chamber 17. For example, certain formulations of steel provide a "gasy" melt, because of the presence of oxygen and argon. This process may be taken of the evolution of the gas during the atomizing process to help maintain controlled pressure within the chamber. The oxygen generally combines with carbon present in the melt, during solidification, and come off as carbon monoxide (CO) gas. Normally, of course, the out-gassing of the molten metal is insufficient, in itself, for adequate pressure control, and supplementary quantities of inert gas are introduced by the regulator 34.

Usually, it is desirable to purge the atomizing chamber 17 prior to the commencement of the atomizing operation. Typically, this can be done by introducing argon into the interior of the atomizer housing, expelling the atmospheric air, and then sealing over the crucible opening 21 with a destructible film, such as aluminum foil. When the molten metal subsequently is poured into the crucible, the seal is instantly broken. However, the molten metal itself functions as a seal, as long as a quantity thereof remains in the crucible 19.

The production of water-atomized metal powders in accordance with the invention can be carried out in a manner to achieve oxide levels which have never before been achieved in a water atomizing process. In this respect, it is possible to achieve water atomized powder, the oxygen content of which is far below the 0.25 percent at which it becomes necessary to perform further, costly reduction processes to condition the metal properly for many end uses. Even so, the powder produced in accordance with the invention should be handled at subsequent stages in an appropriate manner so that the dried powder available for ultimate utilization in the formation of wrought products or compacts, remains well below the 0.25 percent oxygen content level.

As illustrated in FIG. 1, the receiving tank 10 has its outlet 12 connected to a suitable separating device usually of a gravity type, designated by the numeral 35. Periordinically (or continuously, if desired) water and entrained particles are flowed or pumped to the separator 35, which is adapted to remove low density impurities such as slag, furnace refractories, etc. The impurities are discharged at 36, and the mixture of water and higher density particles is suitably drained at 37 to remove most of the water content. Thereafter, the still wet powder containing from 1 percent to as much as 15 percent–20 percent water, is taken directly to a drying facility 38, where the remaining water is removed. Advantageously, the drying facility 38 is a vacuum dryer, from which the air is first exhausted, (eliminating oxygen), and then the powder is heated while retaining a vacuum. This results in a dried powder with minimum oxide gain from its as-atomized, wet condition. Alternatively, the powder may be dried in a reducing atmosphere.

Referring now to the modified embodiment shown in FIG. 4, the receiving tank 10 is somewhat smaller than receiving tank 10 in the FIG. 1 embodiment, thus holding a smaller amount of cooling liquid, because with the arrangement in the modified showing of FIG. 1, it is not necessary to have quite such a large body of turbulent receiving and cooling liquid 11, although it should be understood that the embodiment of FIG. 4 can accommodate any size body of cooling liquid with any size receiving tank 10.

Suitably mounted on the receiving tank 10 is an atomizer housing 113, similar in construction to that of housing 13 in the FIG. 1 embodiment, with housing 113 also constituting a sealed enclosure 17 with a water leg 118 of substantially smaller cross-section than leg 18 of the FIG. 1 embodiment which extends up to atomizer housing 113.

As in FIG. 1, housing 113 is provided with an opening 114 at the top for the introduction of molten metal to the atomization thereof, with a similar opening 15 in the upper portion thereof for admitting inert gas. A discharge outlet 116 is provided in the lower extremity of water leg 118, below the water level in the retaining tank 10. As indicated in FIG. 4, the discharge opening 116 of the atomizer housing is of smaller dimensions than the upper portions thereof constituting the atomizing chamber 17.
However in this embodiment the plurality of pairs of intersecting water jets, to be described in detail below, are arranged to intersect the molten metal stream and the atomized particles therefrom immediately adjacent the top of water leg 118. With such an arrangement, therefore, there is continuous turbulent liquid-particle contact throughout the length of water leg 118, thus further reducing the temperature of the atomized particles, and as a consequence, minimized oxide formation at the liquid particle interface.

Furthermore, with increased liquid volume in this embodiment and because the liquid jets are arranged so closely adjacent the top of water leg 118, the comingled particles and liquid are actually directed downwardly into water leg 118 itself. Thus, liquid flow volume through water leg 118 is a high-inertia flow controlling the entire cross-sectional area of water leg 118 through the entire length thereof. As a result, even though there is a high vacuum created in chamber 17 which would normally cause a back-up in water leg 118 of several feet in response to that vacuum, the high-inertia flow providing rapid, turbulent, unimpeded passage, in accordance herewith, will substantially overcome this problem. Accordingly, with the arrangement as described herein, a long unwieldy water leg is not required even though increased volume issuing from the jets would ordinarily require it.

This is particularly desirable because it has been found that increased retention time in an extended water leg will cause impeded flow and a substantial increase in oxide formation on the surfaces of the particles. Also, because outlet 116 is restricted in relation to volume flow through leg 118, no back-up from tank 10 will result.

As in the FIG. 1 embodiment, at the top of the atomizer housing 113, a receiving crucible 19 is disposed for receiving, when the system is in operation, a body of molten metal 20. The crucible 19 has an opening 121 in its bottom wall, which communicates with the interior of the atomizer housing 113 and provides for the gravity discharge assisted by the vacuum therein of molten metal in a solid stream.

In the FIGS. 4-6 embodiment of the invention, three or more sets of liquid spray jets may be provided in the atomizing chamber 17, disposed to act in rapid sequence upon the descending stream of molten metal. As is shown in FIG. 5, a first pair of water discharge nozzles 22, 23 is disposed symmetrically on opposite sides of the descending, coherent stream of molten metal 124. As in the FIG. 1 embodiment, the nozzles 22, 23 are directed downward and inward at an angle of within the range of between about 15°-30° from the vertical, and are arranged to direct controlled jets of atomizing water into intersecting relationship with each other and the molten metal stream 124.

As is discussed above, it is important to derive, in the metal atomizing stage, metal particles which are fine in size and are angular and irregular in configuration. This is achieved through the use of thin, flat, solid streams 25, 26 of atomizing water, ejected from the nozzles 22, 23 at high pressure. As in the FIG. 1 embodiment, the atomizing streams 25, 26 are ejected under pressures within the range of between about 500-1,000 psi (pounds per square inch) or more. These nozzles are arranged in the manner of relationship to each other and to the stream of molten metal, with the nozzles being of substantially the same configuration as in FIG. 1, in order to provide the desired solid, flat streams of water, to intersect with each other and with the molten stream at point 27.

As in the arrangement in FIG. 1, this embodiment will produce, in accordance herewith, metal particles substantially all of which are minus 40 mesh in size and preferably minus 80 mesh, which means that almost all the particles produced would pass through a mesh screen within the range of between about minus 40-80 mesh (U.S. Sieve Series, A.S.T.M. Specification E-11-61).

With the arrangement in FIGS. 4-6 in order to obtain atomized metallic particles in the desired non-oxidized form, two or more additional intersecting sets of water nozzles 28, 29 and 42, 44 are arranged to direct water streams 30, 31 and 46, 48, respectively at intersecting points 32, 30, respectively. The additional water streams are brought as close up to the atomizing streams 25, 26 as practicable, as in FIG. 1, without causing interference with the action of the atomizing streams. In practice, in an atomizing apparatus of the general dimensions and configurations described, the first additional streams may intersect at a point from as close as 1/4 inch to as far as 2 inches below the intersecting point 27 of the atomizing streams, with a more typical spacing being approximately 3/4 inch.

As is shown in FIG. 5, the water streams 46, 48 are arranged to intersect at point 50, the same distance dimensions as the relationship between intersecting point 32 and intersecting point 27.

As in the embodiment of FIG. 1, the objective in the case of the first and second quenching streams is to envelop the just-atomized particles in a substantial volume of water, accompanied by violent turbulence. In this connection, the intersections of the various pairs of atomizing and quenching streams of water with the stream of molten metal and the atomized particles therefrom takes place adjacent the top of water leg 118, in order to direct the turbulent co-mingling of the atomized metallic particles with the jet action of the atomizing and quenching water streams directly into water leg 118, in order to enhance the turbulence of this co-mingling.

As stated above, with the outlet opening 116 substantially restricted, relative to the volume of water flow, there is a high-inertia flow from the nozzles to help control the vacuum in the atomizer housing 113 and prevent water back-up in the leg 118. In this connection, assuming a pilot size operation with the outlet opening 116 being of a size of 1/2 inch X 4 inches, the volume of water flow will be within the range of between 70-80 gallons per minute. Such an arrangement will accommodate a molten metal stream flow from crucible 19 of approximately within the range of between about 60 pounds per minute — 120 pounds per minute of metal.

It should be understood, in this connection, that it is within the purview of this invention that more than two pairs of second stage jets may be utilized, depending upon a variety of factors including the size of the operation, the nature of the metal being atomized, the size of the water leg and so forth. The only limitation required is that the jet provide sufficient volume of water under a pressure appropriate for maintaining the high-inertia water flow over the entire cross-section of outlet 116 of water leg 118.
After exposure of the metal particles to the additional stage of streams 30, 31 and 46, 48, it is desirable to follow immediately with a further cooling stage. In this connection, the quenched particles are flowed downward through the water leg 118 and are ejected out through discharge opening 116. In this connection, with the arrangement herein and the increased volume of water utilized, the water leg 118 may be of substantially shorter dimensions, as is shown in FIG. 4.

As in FIG. 1, also, the water issuing from the discharge nozzle 116 has sufficient forward discharge velocity to maintain a desired condition of substantial turbulence within the water body 11. Again, supplementary agitation of the main body 11 of cooling water may be provided, if desired. Also, the water utilized for quenching and cooling may be heated to reduce its content of dissolved oxygen, in order to further reduce the formation of surface oxides on atomized metallic particles.

As was stated above, it is one advantageous feature of the embodiment of the invention in FIG. 4, because of the restricted outlet 116 of water leg 118 relative to the water flow volume, to provide a high-inertia flow from the nozzles to help control the water level in atomizer housing 113. Additionally, pressure in atomizing chamber 17 and atomizer housing 113 is controllably increased by means of a supply (not shown) of inert gas, such as argon, for example, which is fed in through conduit 33, through flow pressure regulator 34, as in the FIG. 1 embodiment. Regulator 34 will be set at a level which will retain a column of water in water leg 118, which, even allowing for substantial surface turbulence, will usually provide sufficient clearance below the water jets to avoid interference with their action. Again, the out-gassing of the metal itself may be utilized to advantage in controlling the pressure in the sealed atomizer chamber 113.

Thus, it is important, in accordance herewith, to maintain the proper relationship between the volume and the pressure of the water being utilized, the metal flow rate, the gas content of the metal being utilized, the amount of inert gas necessary to control the water level in the leg 118, and the size of opening 116 at the bottom of water leg 118 (the smaller the opening, the greater the jet effect of flow through the water leg 118).

It should be understood, in accordance herewith, that the introduction of the inert gas should be used at least at the start of the process in order to avoid explosions, whether or not inert gas is continued to be used in order to control the vacuum in the atomizer housing 113.

The ability to produce water-atomized powders, of iron, steel, and other materials, with the extremely low oxygen content enabled by the present invention, permits extraordinary economic advantages to be realized. By way of example, an iron or steel powder thus produced, having an oxygen content well below 0.25 percent, can be used directly in the manufacture of strip and wrought products, without undergoing special reducing processes. Thus, iron and steel powders produced in accordance with the invention generally will have no more than an extremely thin oxide film at the surface, as is evidenced by a light gray cast. This thin film can be flashed off if necessary, quickly and economically in a reducing atmosphere after compaction of the powder into a green strip and while the green strip is being conveyed through a furnace for heating to temperatures suitable for hot rolling. More conventional powders, having high oxygen content if water atomized, typically will have to be reduced separately, prior to formation of the green compact. The much heavier oxide coating of conventionally water atomized particles is characterized by a dark gray or black surface coloration (reflecting an oxygen content of 0.8 percent or more), in the case of iron and steel particles.

In some cases, as where it is desired to produce wrought products from iron powders having a high carbon content, it may be necessary to utilize a preliminary tempering heat treatment to soften the high carbon powder sufficiently to carry out the compacting operation. In such cases, the cost of a separate heating operation cannot be avoided. However, important advantages are still realized, in that it is possible to carry out the heating step at comparatively low temperatures (1,000°-1,200°F.) without significantly changing the composition of the metal. In this respect, if there is substantial oxygen present with high carbon powder, there is a tendency for the carbon and oxygen to react, forming CO and CO₂. Carbon may also react with hydrogen at temperatures sufficient to remove the oxygen. This results in an undesirable composition change, where a high carbon product is desired.

The avoidance of a separate reducing step, made possible by the atomizing procedures of the invention, also has important ramifications in respect to arc grain formation. Thus, by the extremely rapid atomization, quenching and cooling accomplished in accordance with the invention, exceedingly fine grain sizes within the particles are realized. This is highly desirable for many applications. If a separate reducing operation is required, as in conventional processes, a grain growth (within the much larger atomized particles) necessarily results, through an irreversible process. Materials manufactured in accordance with the present invention, inherently will have especially fine grain sizes. If, for some reason, larger grain sizes are desirable, they may be achieved through subsequent appropriate heat treating processes. However, the reverse is not true of conventionally produced material.

One very important advantage derived from this invention in avoiding a reducing step after atomization resides in the ability to compact the powder into strip, rods, forging blanks, etc., while the powder remains in its internally stressed, “as atomized” condition. The high energy state of the internally stressed atomized particles provides for the achievement of final products of the desired density and enhanced coherency.

By way of examples, typical metal powders produced in accordance with the invention may be of the following representative analysis:

**I. LOW CARBON STEEL, DRIED IN ALCOHOL**

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.05%</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.24</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.12</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.031</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.007</td>
</tr>
<tr>
<td>Oxygen</td>
<td>0.136</td>
</tr>
</tbody>
</table>

**II. LOW CARBON STEEL, DRIED IN ALCOHOL**

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.038%</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.26</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.14</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.030</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.008</td>
</tr>
</tbody>
</table>
Copper (0.01)
Oxygen (0.168)

III. HIGH CARBON STEEL, DRIED IN ALCOHOL

Carbon (0.39)
Manganese (0.20)
Silicon (0.03)
Phosphorus (0.005)
Copper (0.09)
Nickel (0.05)
Chrome (0.37)
Oxygen (0.18)

IV. STAINLESS STEEL (RAZOR BLADE GRADE), VACUUM DRIED

Carbon (0.60)
Chrome (12.63)
Manganese (0.71)
Silicon (0.28)
Phosphorus (0.01)
Oxygen (0.21)

V. NICKEL ALLOY, VACUUM DRIED

Nickel (29)
Cobalt (17)
Iron (54)
Oxygen (0.16)

VI. NICKEL ALLOY, VACUUM DRIED

Nickel (79)
Molybdenum (2)
Iron (19)
Oxygen (0.09)

VII. COPPER ALLOY, OPEN DRIED

Copper (84.5)
Zinc (15.43)
Iron (0.005)
Lead (0.003)
Oxygen (0.09)

Samples I, II and III reflect a final oxygen content of 0.18 percent for high carbon steel and less than 0.17 percent for low carbon steel. Powders of corresponding analysis, water atomized by conventional procedures, would reflect an oxygen content of well over 0.25 percent after drying and would surely require a separate reduction procedure for most end uses. Sample IV reflects an oxygen content of 0.21 percent after drying, whereas material of similar analysis, atomized conventionally, would typically have an oxygen content of over 0.25 percent. Sample V is a nickel-steel alloy powder reflecting an oxygen content of 0.16 percent. Samples VI and VII are nickel and copper alloy powders reflecting oxygen contents of 0.09 percent. In all samples, the techniques of the invention have enabled a significantly lower oxygen content to be realized in the dried powders.

The oxygen contents reflected in the foregoing analyses have not, heretofore, been attainable or even approached, using conventional water atomizing techniques. Considering oxygen content alone, it has been possible to achieve such low levels using inert gas as the atomizing medium. However, atomizing processes using inert gas as the operative medium have fundamental disadvantages which more than compensate for their ability to achieve low oxide production. For one, the production rate is extremely slow; for another, the powder configuration is essentially spherical, because of the slow rate of heat transfer; and, for another, the economics of atomizing with inert gas are quite unattractive.

The apparatus of the present invention is ideally suited for production on an industrial scale, using equipment of a practical, trouble-free nature, which can be set up and operated on an economic basis. It should be understood, of course, that the foregoing description of the invention is intended to be representative only. Reference should be made to the following appended claims in determining the full scope of the invention. In the foregoing specification, and in the claims, the term "iron" shall be considered to include steel, wherever the context admits thereof, and the term "steel" shall be considered to include alloys containing 50 percent or more iron by weight.

What is claimed is:

1. Apparatus for producing metal powder, comprising
   a. an atomizer housing forming an enclosed chamber;
   b. a receiving vessel for containing a body of cooling water;
   c. means mounting said housing at least partly within said vessel whereby the lower end of the housing is immersed in and sealed by said body of cooling water;
   d. said housing including an atomizing chamber portion spaced above the surface of the body of water;
   e. means to introduce molten metal into the upper portion of the housing;
   f. means to direct high velocity streams of water at said molten metal streams in said atomizing chamber;
   g. said housing including a sealed water leg portion connecting said atomizing chamber with said body of water and providing a passage for the ejection of water and atomized particles from said housing;
   h. means for maintaining said atomized particles in contact with water under conditions of violent turbulence, substantially continuously from the atomization of said particles by said high velocity streams to the ejection thereof into said body of cooling water.

2. An apparatus according to claim 1, further characterized by said means to direct high velocity streams including
   a. a first pair of water nozzles disposed to issue atomizing water streams in intercepting relation to each other and to the stream of molten metal; and
   b. a second pair of water nozzles disposed to issue quenching water streams to intersect each other directly below and closely adjacent to the interception of said metal by atomizing water streams.

3. An apparatus according to claim 2, further characterized by
   a. said atomizing water streams being of flat, thin, solid configuration and being issued at a pressure of about 500 psi or greater; and
   b. said quenching water streams being issued at a pressure of about 100 psi or greater.

4. An apparatus according to claim 1, further characterized by
   a. means for introducing gas at a controlled flow rate into said atomizing chamber to control the water level in said water leg.

5. An apparatus according to claim 1, further characterized by
   a. the lower end of said housing including a nozzle-like discharge portion of reduced cross-section, as
compared to the cross-section of the atomizing chamber, for the discharge of water from said water leg into said body of cooling water at relatively high velocity;

b. said nozzle-like discharge portion being disposed at a large angle to the vertical to promote a horizontal flow, away from said discharge portion, of discharged water and entrained atomized particles.

6. Apparatus for atomizing molten metal to a powder having irregular, angular particles predominantly of minus 40 to plus 325 mesh and containing less than 0.25 percent oxygen by weight, comprising
a. an atomizer housing forming an enclosed chamber;

b. a receiving vessel containing a body of cooling liquid;

c. a water leg extending from said housing to said vessel, with the upper portion of the walls of said leg forming a continuation of the walls of said atomizer housing and the lower end of said leg having a restricted outlet immersed in and sealed by said body of cooling liquid;

d. means for introducing a molten metal stream into the upper portion of said enclosed chamber;

e. first means for directing high velocity streams of cooling liquid at said molten metal stream closely adjacent the upper portion of said water leg in said enclosed chamber for the atomization of said molten metal stream into particles;

f. second means for directing high velocity streams of cooling liquid at said atomized molten metal stream closely adjacent the upper portion of said water leg for maintaining said atomized particles in contact with cooling liquid under conditions of violent turbulence;

g. said first and second means being positioned for impingement downwardly into said water leg for substantially continuously maintaining violent turbulent contact, and

h. said water leg providing a continuously substantially unimpeded passage for the injection of cooling liquid and atomized particles from the upper portion through the outlet thereof.

7. Apparatus as recited in claim 6, further characterized by said first means to direct high velocity streams including
a. a pair of nozzles disposed to issue atomizing cooling liquid streams in intercepting relation to each other and to the stream of molten metal, and

b. said atomizing cooling liquid streams being of flat, thin, solid configuration and being issued at a pressure of at least 500 pounds per square inch at an angle within the range of between about 15°-30° from the axis of said streams of molten metal.

8. Apparatus as recited in claim 6, further characterized by said second means for directing high velocity streams of cooling liquid including
a. at least two pairs of spaced apart nozzles, with each pair being disposed to issue cooling liquid streams to intersect with each other directly below and closely adjacent to the interception of said metal by said atomizing cooling liquid streams, and

b. said pairs of spaced apart intersecting cooling liquid streams being issued at an angle within the range of between about 15°-30° from the axis of said stream of atomized metal particles.

9. Apparatus as recited in claim 6, further characterized by
a. means for introducing inert gas at a controlled flow rate into said atomizer housing for maintaining a non-explosive atmosphere therein and for partially controlling the cooling liquid level in said water leg.

10. Apparatus as recited in claim 6, further characterized by
a. the lower end of said water leg including a nozzle-like discharge portion of reduced cross section, as compared to the cross section of the upper portion of said leg.

11. Apparatus as recited in claim 6, further characterized by said first and second means to direct high velocity streams including
a. means to direct combined cooling liquid streams under sufficient pressure to maintain high velocity flow of cooling liquid and atomized particles over the entire transverse extent of the outlet of said water leg.

12. Apparatus as recited in claim 10, further characterized by said first and second means to direct high velocity streams including
a. means to direct combined cooling liquid streams under sufficient pressure to maintain high velocity flow of cooling liquid and atomized particles over the entire transverse extent of the outlet of said water leg.

13. Apparatus for atomizing molten metal to a powder having irregular, angular particles predominantly of minus 40 to plus 325 mesh and containing less than 0.25 percent oxygen by weight, comprising
a. a sealed chamber;

b. a receiving vessel containing a body of cooling liquid;

c. means providing flow communication between said chamber and said body of cooling liquid;

d. means for introducing a molten metal stream into said sealed chamber;

e. means for intercepting said molten metal stream sequentially with a plurality of opposed cooling liquid streams for atomizing said molten metal stream and for cooling the individual particles of said atomized molten metal stream;

f. said interposing means being arranged to direct said plurality of opposed cooling liquid streams into and through said flow communication means, and

g. means for controlling said intercepting means to provide high velocity unimpeded flow across the entire cross section of said flow communication means from said chamber to said body of cooling liquid.

14. Apparatus as recited in claim 13, further characterized by
a. said flow communication means being a water leg of gradually reduced cross section from said chamber to said body of cooling liquid.

15. Apparatus as recited in claim 14, further characterized by
a. said intercepting means being a plurality of pairs of nozzles disposed to issue cooling liquid streams in intercepting relation to each other and to the stream of molten metal, and

b. said cooling liquid streams being of flat, thin, solid configuration and issued at an angle of between
about 15–30 degrees from the axis of said stream of molten metal.

16. Apparatus as recited in claim 13, further characterized by
   a. means for introducing inert gas at a controlled flow rate into said sealed chamber for preventing cooling liquid flow from said body of cooling liquid into said flow communication means.

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