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Reed et al.

[11] **3,752,611**

[45] **Aug. 14, 1973**

[54] **APPARATUS FOR PRODUCING METAL POWDER**

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[73] Assignee: **Republic Steel Corporation**, Cleveland, Ohio

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[21] Appl. No.: **175,407**

Related U.S. Application Data

[62] Division of Ser. No. 834,368, June 18, 1969, Pat. No. 3,655,837.

[52] **U.S. Cl.** **425/7,** 425/10, 264/6

[51] **Int. Cl.** **B22d 23/08**

[58] **Field of Search** 425/7, 10, 6; 264/12, 6, 5, 11, 7

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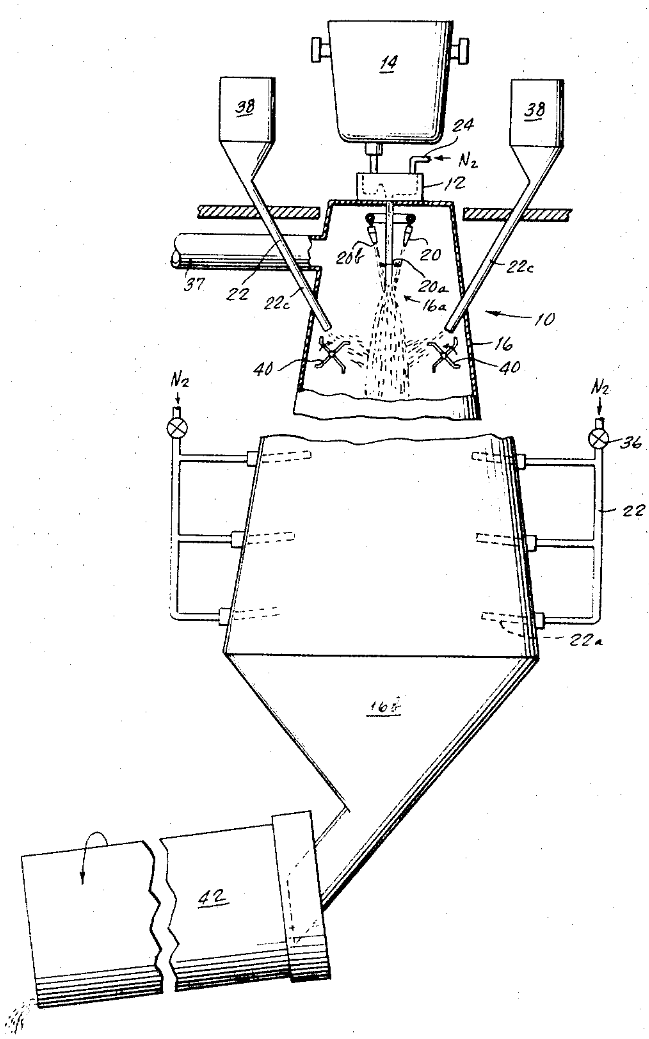
[57] **ABSTRACT**

Apparatus and process for producing a powder from molten metal by forming a flowing stream of metal from a ladle through a regulating tundish and into an atomizing chamber having an atomizing zone and a collecting zone. An atomizing gas is directed through an annular nozzle or series of annularly disposed gas jets against the stream of molten metal to form metal particles, the gas exiting from the nozzle or jets at supersonic velocity. Matter is injected against the particles prior to atomization and/or between the atomizing and collecting zones of the chamber to cause particle agglomeratio to produce particles of irregular shape and to cool the particles. The injected matter may be an inert gas or fine particles of metal powder. The particles settle in the collecting zone where they are subsequently cooled and transported for further processing.

6 Claims, 9 Drawing Figures



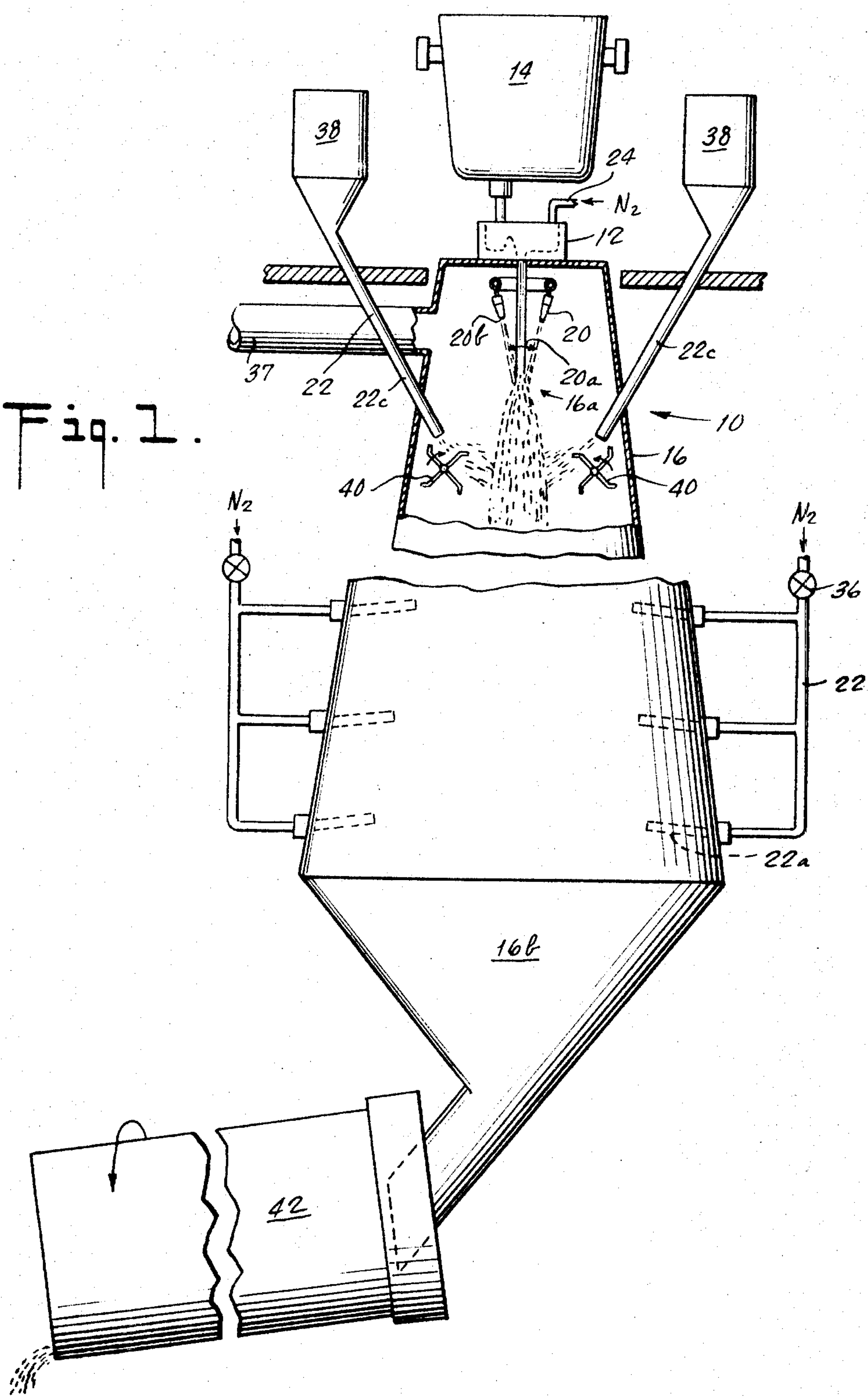

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Fig. 1.

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Fig. 2.

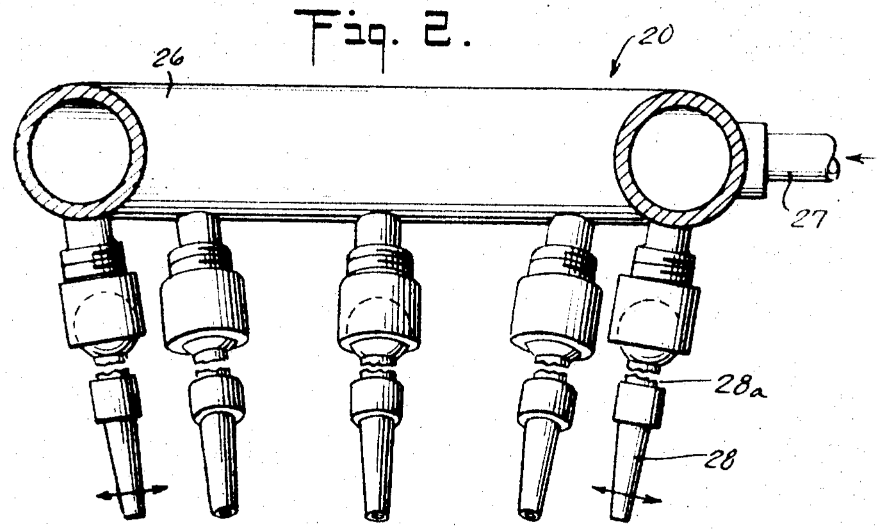
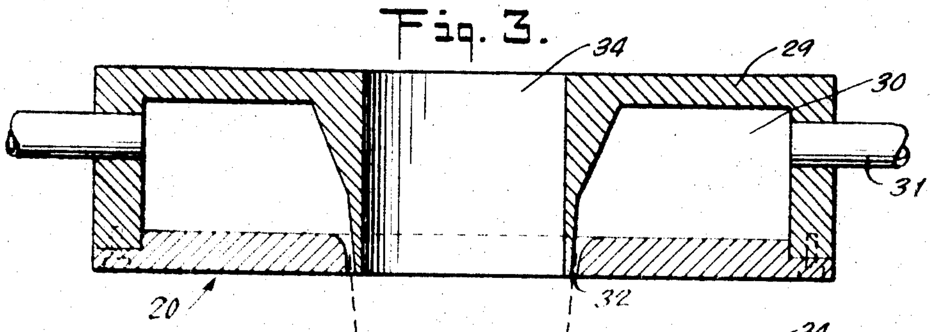


Fig. 3.



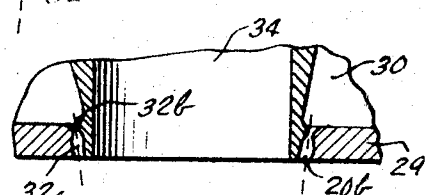


Fig. 4.

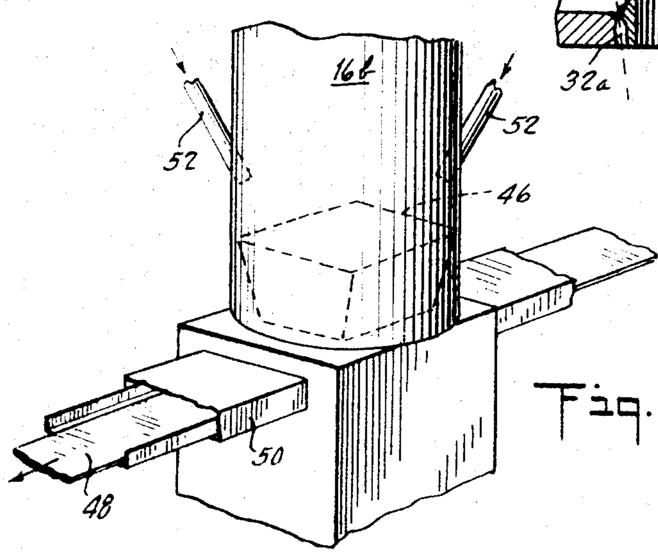


Fig. 9

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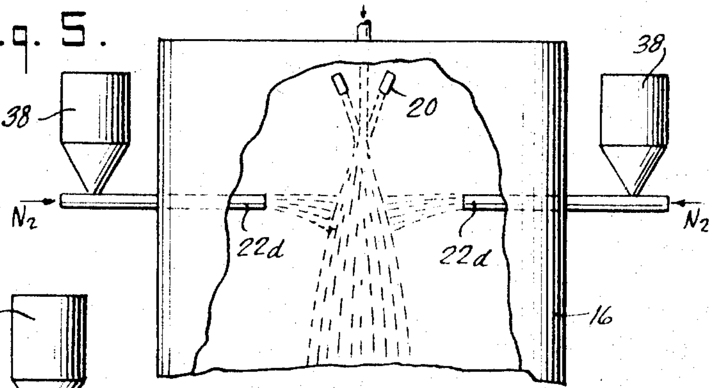
Fig. 5.



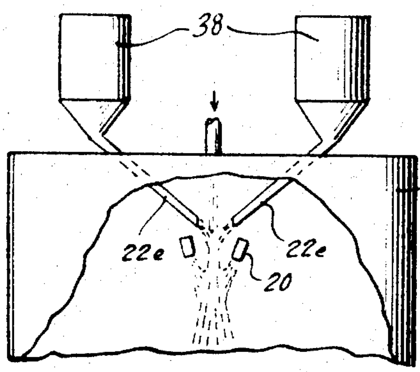


Fig. 6.

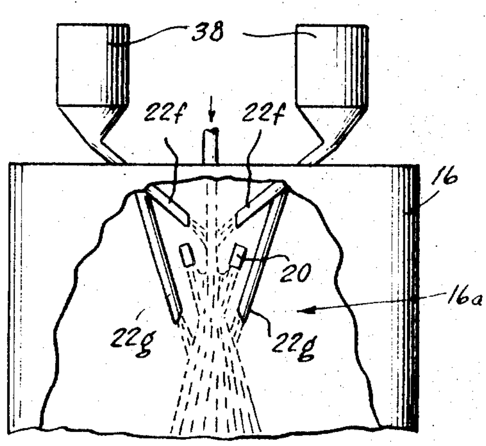


Fig. 7.

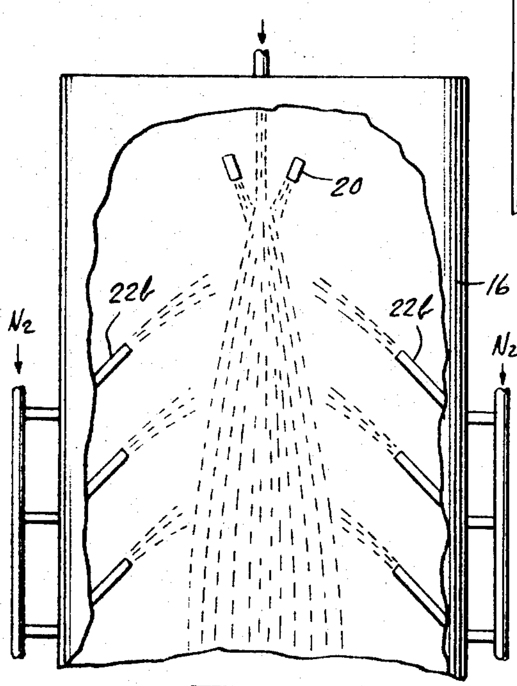


Fig. 8.

APPARATUS FOR PRODUCING METAL POWDER

CROSS REFERENCE TO RELATED APPLICATION

This application is a division of my co-pending application Ser. No. 834,368, filed 18 June 1969 for APPARATUS AND PROCESS FOR PRODUCING METAL POWDER, now Pat. No. 3,655,837 issued 11 Apr. 1972

BACKGROUND OF THE INVENTION

The present invention relates to apparatus and process for producing metal powder. In particular, the invention involves the manufacture and preparation of metal powder by injecting an inert gas or fine particles of metal powder into the flow of molten metal and/or into the flow of metal particles formed by the atomization of molten metal.

It is known in the art to produce atomized powder which may be compacted into strip, sheet, plate, or finished items such as gears. In general, atomization is a process which involves the disintegration of a stream of molten metal into individual droplets which are subsequently solidified. An atomizing jet of fluid, e.g., water, air or inert gas, is directed toward the molten stream in order to break up the stream into fine particles, which are cooled and solidified.

There are several problems inherent with conventional atomization powder-producing processes. If water or air is used as the atomizing fluid, the resulting particles may contain an oxide inclusion and have an oxide coating on their outer surface. If inert gas is used as the atomizing fluid, the particles formed may have a regular spherical shape which does not lend itself to good compaction of the powder. Although water will produce the desired irregularly shaped particles, the high oxide content of the product negates this advantage. Furthermore, the hot particles formed by inert gas atomization which accumulate on the bottom of the atomization chamber form a sintered mass or cake which must then be ground to obtain particles of usable size for subsequent consolidation into a compacted product. Frequently, the sintered mass or cake is not sufficiently friable to be readily ground without destruction of basic particle shapes. It is therefore desirable to provide a process and apparatus which efficiently produces a metal powder suitable for subsequent compaction.

BRIEF SUMMARY OF THE INVENTION

An object of the present invention is to provide a method and accompanying apparatus for atomizing molten metal into a powder comprised of particles of irregular shape and lacking an oxide coating. It is another object of the invention to produce a metal powder which agglomerates but which can be readily ground into particles having optimum compacting properties. Still another object is to provide a means for dissipating sufficient heat from the atomized metal to prevent fusion of the particles before they can be collected from the base of the atomizing chamber. A further object is to provide a nozzle for an atomizer which does not readily become encrusted and hindered in its operation.

To these and other ends, the instant invention contemplates the process of forming a flowing stream of molten metal, e.g., low-carbon, aluminum-killed, drawing-quality steel, directing an atomizing inert gas into an atomizing zone against the metal to produce particles, injecting matter against the particles prior to atomization and/or after they emerge from the atomizing zone to agglomerate them into irregular shapes and to cool the particles, and collecting the particles in a collecting zone.

In accordance with the invention, the molten metal is poured from a ladle into a tundish, which causes a smooth steady stream of molten metal to be introduced into an atomizing chamber. There an inert atomizing gas is directed against the molten metal in an atomizing zone to separate the molten stream into discrete particles. The gas is directed toward the molten stream by an annular nozzle means, e.g., a single annular nozzle or a series of jets annularly disposed about the flow of molten metal and constituting a nozzle, which may be of the converging-diverging type to produce a stream of gas which leaves the nozzle at supersonic velocity. An included gas angle of about 10° to 12° is desirable to prevent the nozzle from clogging if a converging-diverging type of annular nozzle is employed, but an included gas angle of less than 40° is preferable when separate converging-diverging jets annularly spaced are used. The gas pressure should be about 100 to 150 pounds per square inch and the gas should flow at a rate of about 1,200 cubic feet per minute for a molten flow of 150 pounds per minute. The diameter of the nozzle (the diameter of the annulus defined by either a single annular pipe or the like forming a nozzle or a series of annularly disposed jets forming a nozzle) is preferably about three to four times the diameter of the molten metal stream.

Before and/or after the molten metal has been atomized into particles, secondary matter is injected into the atomizing chamber against the metal to cause the particles to agglomerate into irregular shapes and to cool them. This injected matter may be either an inert gas or fine particles of powder, e.g., as produced from atomization and recirculated, or both inert gas and powder. If inert gas is employed, it may be directed toward the particles between the atomizing and collecting zones of the atomizing chamber in a direction substantially transverse to or against the flow of particles. If fine particles of powder are employed, the particles may be contained in a hopper and directed toward or with the flow of molten metal or atomized particles by a nozzle or an impeller. The ratio of injected fine particles to atomized molten metal is preferably about 1:1 by weight.

Means including a conveyor are provided near the base of the atomizing chamber for collecting the produced powder before it can become sintered or caked. A rotary cooler may also be employed near the base of the atomizing chamber.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an elevational view, partly in section, of representative apparatus in accordance with the present invention.

FIG. 2 is an enlarged partial view of one form of atomizing nozzle of the invention.

FIG. 3 is an enlarged sectional view of another form of atomizing nozzle used in the invention.

FIG. 4 is a partial sectional view of still another form of atomizing nozzle used in the instant invention.

FIG. 5 is a broken view of another form of apparatus of the invention.

FIG. 6 is a broken view of still another form of apparatus of the invention.

FIG. 7 is a broken view of a further modification of the apparatus.

FIG. 8 is a broken view of yet another embodiment of the apparatus of the invention.

FIG. 9 is a perspective view of a means for removing metal powder from the atomizing chamber in accordance with the present invention.

DETAILED DESCRIPTION

Referring to FIG. 1 of the drawings, there is shown representative atomizing apparatus **10** in accordance with the present invention. The apparatus includes a tundish **12** for receiving molten metal, e.g., low carbon, aluminum-killed, drawing-quality steel (termed herein AKDQ steel), discharged from a ladle **14** and providing a smooth flowing stream of molten metal. There is provided an atomizing chamber **16** having an atomizing zone **16*a*** into which the stream of molten metal from the tundish **12** flows for the purpose of atomization of the metal into powder. A collecting zone **16*b*** within the chamber **16** receives the produced powder. Disposed within the atomizing chamber **16** is an atomizing nozzle **20** for directing an atomizing gas against the stream of molten metal to atomize the metal into powder. A series of secondary nozzles **22** is provided for injecting additional matter into the chamber **16** against the flow of molten metal and/or the produced metal powder in order to agglomerate and cool the metal powder so that it is suitable for further processing into metal plate, sheet, or strip, for example.

In accordance with the invention, molten metal contained within the ladle **14** is preheated to a sufficient temperature to ensure proper pouring. The molten metal is then discharged from the bottom of the ladle **14** into the tundish **12**, which should be heated to a temperature of about 2,800° F prior to pouring (in the case of AKDQ steel) in order to prevent a frozen melt. The tundish regulates the flow of molten metal from the ladle **14** into the atomizing chamber **16**. The tundish **12** may be fabricated from a refractory material, e.g., alumite (93% Al_2O_3), which has been cast in a mold and fired slowly to about 1,400° F, e.g., prior to use. Although a tundish **12** having one feeding nozzle is illustrated, it is possible to employ a tundish which will provide multiple streams of molten metal.

Associated with the tundish **12** is an inlet pipe **24** for allowing an inert gas, e.g., nitrogen gas, to be introduced into the tundish. This inert gas prevents oxides from forming in the molten metal prior to atomization. In order to produce a suitable metal powder, the atomizing chamber **16** should also be purged with inert gas for about a 30-minute period prior to atomization at a gas flow rate of about 190 cu. ft. per minute for a chamber volume of about 315 cu. ft. This inert gas atmosphere reduces oxidation of the produced metal particles.

After the molten metal has been formed by the tundish **12** into a smooth flowing steady stream, it is permitted to enter the atomizing zone **16*a*** of the atomizing chamber **16** where the molten metal at a temperature of about 2,950°–3,000° F is transformed into metal particles by means of a stream of inert gas, e.g., nitrogen preferably of about 99.995 percent purity, directed downwardly from an atomizing gas exit zone **20*b*** against the molten metal stream by atomizing nozzle **20**. If a nozzle **20** of the converging-diverging type is employed to create a supersonic flow of gas, the gas exit zone **20*b*** will be in an area located immediately beyond the restriction but within the orifice of the nozzle **20** and is the point where the atomizing gas is at its greatest velocity. The atomizing gas will not maintain its supersonic velocity at the point of intersection with the stream of molten metal. Several designs of atomizing nozzles **20** have been found suitable for employment in the present apparatus. In any case, the nozzle **20** should provide an included gas angle (designated **20*a*** in FIG. **1**) with the orientation of each port of the nozzle at an angle from the vertical equal to one-half the included gas angle and directed toward the central axis of the atomizing nozzle **20**. The gas from the nozzle **20** atomizes the molten stream of metal into particles of fine size, which thereafter cool and which may be collected at the bottom of the atomizing chamber **16**.

It is necessary to use a nozzle **20** with an included gas angle sufficient to prevent coarse, hot particles from striking back at and tending to stick to the nozzle. Such sticking particles would bridge the nozzle and result in a partial or complete reduction of the stream of molten metal. An atomizing gas pressure of about 100 to 150 lbs. per square inch with a flow rate of about 1,200 to 1,500 cu. ft. per minute for a molten metal flow of about 150 pounds per minute or about 8 to 10 cu. ft. of gas per pound of molten metal acted upon is preferred. Lower gas pressures produce extremely coarse particles which form a sintered mass at the bottom of the atomizing chamber **16** (due to heat retention in the particles), while higher gas pressure in combination with a high included gas angle **20*a*** results in more particles striking back at the nozzle **20** and thus causing blockage of the nozzle. Furthermore, it is preferable that the diameter of the atomizing nozzle **20** be approximately three to four times the diameter of the molten metal stream flowing from the tundish **12** in order to reduce the pressure within the nozzle and the tendency of the metal particles to strike back at the nozzle.

FIGS. **2** through **4** show various forms of atomizing nozzles **20** which may be used to direct the inert gas against the stream of molten metal. In FIG. **2**, the atomizing nozzle **20** comprises an annular pipe **26** associated with a source of inert gas through an inlet pipe **27**. The annular pipe **26** may alternatively be comprised of quadrants separated by partitions (not shown); in that case, four inlet pipes **27** would be required to supply inert gas to the nozzles **20**. Such an alternative arrangement allows greater control over the flow of inert gas and over the deviation of the stream of atomized metal from the vertical. A number of gas jets **28** are disposed equally about the perimeter of the annular pipe **26**. Each gas jet **28** is oriented at a suitable angle from the vertical (up to 20°, for an included gas angle of up to 40°) and points toward the central axis of the annular pipe **26** such that the series of gas jets **28** circumscribes the stream of molten metal and the gas cone therefrom intersects the stream at a distance from the nozzle **20** within the atomizing zone **16*a*** of the atomizing chamber **16**. The optimum dimensions for a series of gas jets **28** have been found to be an included gas angle of about 30° and a nozzle effective diameter of about 3 ½ in. for a molten stream of metal approximately nine-sixteenths in. in diameter. Ungrindable coarse material is at a minimum when the effective diameter of the jets **28** is that value. If the diameter is either too large or too small the amount of +8 mesh product (large size coarse material which generally cannot be ground finer without flattening or destroying particle shape using conventional grinding equipment) will increase irrespective of an increase in the injection rate of secondary matter. A smaller diameter nozzle also presents a difficulty in assuring proper alignment between the tundish nozzle and the center of the series of jets **28.** Each gas jet **28** may be formed from copper tubing (**28***a*) or the like and may be flexible so that the included gas angle and effective diameter of the nozzle might be easily adjusted by bending the tubing. The gas jets **28** may be provided with a ball-and-socket connection so that the angle of the jets may be easily varied. It is preferred that each jet **28** be of the converging-diverging type so that the gas may exit from the jets at supersonic velocity, as described in more detail below in conjunction with the discussion of the nozzle shown in FIG. **4.**

It has been found that when employing a series of gas jets **28** for atomizing the stream of molten metal, the larger the included gas angle, to some extent the higher the yield of metal powder and the more coarse the powder produced, the powder consisting mainly of agglomerated particles. However, if the included gas angle is too large, it is possible that the nozzle will become encrusted with metal particles and hence hindered in operation.

In FIG. **3** there is shown a conventional atomizing nozzle **20** comprising a housing **29** defining an annular internal chamber **30** associated with a source of inert gas through an inlet pipe **31.** This nozzle **20** has an annular orifice **32** or series of small apetures for directing the inert gas toward the stream of molten metal which passes through an opening **34** in the center of the nozzle **20.** It is preferred that the internal diameter of the annular orifice **32** be approximately 3 inches when the diameter of the molten stream of metal is approximately nine-sixteenths of an inch, as this tolerates to a greater extent misalignment of the tundish **12** and wandering of the stream of molten metal. It has further been found that an annular orifice **32** comprising an annulus of about 0.02 to 0.04 inches is suitable for atomization, unless a high inert gas pressure can be maintained in which case the annular opening may be increased. Such a nozzle has the disadvantage of clogging within a relatively short time due to striking back of the metal particles against the nozzle, especially when the included gas angle **20***a* exceeds 10°.

Referring now to FIG. **4** of the drawings, there is shown a portion of an annular atomizing nozzle **20** similar to the nozzle shown in FIG. **3** but having an annular orifice **32***a* of the converging-diverging type. Such a nozzle includes a restriction **32***b* within the annular orifice **32***a* in order to produce a flow of inert gas which exits the nozzle at the gas exit zone **20***b* at supersonic velocity. An atomizing nozzle **20** possessing the characteristic of supersonic flow is desirable in that it permits more effective usage of the inert gas needed to atomize the stream of molten metal. It is perferred that such a nozzle have an included gas angle **20***a* of about 10° to 12°, when a gas pressure of 100 to 150 lbs. per square inch is employed, in order to prevent clogging of the nozzle. The other dimensions of the nozzle **20** are the same as those for the nozzle shown in FIG. **3.** However, with a converging-diverging type nozzle, the intersection of the stream of inert gas with the flow of molten metal occurs at a point more distant from the nozzle than with a conventional nozzle; thus there is less strike back of the produced metal particles against the nozzle **20** and hence the clogging problem is substantially eliminated. It has been found that a converging-diverging type nozzle may produce a metal powder of intermediate size, e.g., −10 +65 mesh, with a lesser amount of fine and coarse material.

It has been found that a larger included gas angle (**20***a*) may be utilized when individual gas jets (FIG. **2**) are employed rather than an annular nozlzle from a single pipe (FIGS. **3** and **4**). This is probably the result of relieving the vacuum inside the gas cone present with an annular nozzle at higher included gas angles.

In connection with obtaining supersonic flow from a converging-diverging nozzle, the following relationships are useful:

$$T_o/T = 1 + (k - 1/2)M^2 \tag{1}$$

$$P_o/p = [(1 + (k - 1/2)M^2)]^{k/k-1} \tag{2}$$

$$\rho o/\rho = [(1 + (k - 1/2)M^2)]^{1/k-1} \tag{3}$$

$$\frac{A}{A^*} = \frac{1}{M}\left[\left(\frac{2}{k+1}\right)\left(1+\frac{k-1}{2}M^2\right)\right]^{\frac{k+1}{2(k-1)}} \tag{4}$$

$$\frac{\omega}{A} = \sqrt{\frac{k}{R}}\,\frac{p_0}{\sqrt{T_0}}\,\frac{M}{\left(1+\frac{k-1}{2}M^2\right)^{\frac{k+1}{2(k-1)}}} \tag{5}$$

The factors used in the above equations are defined as follows:

T_o = absolute stagnation temperature (in chamber **30**)
T = absolute temperature at any given location in the nozzle
p_o = stagnation pressure (in chamber **30**)
p = pressure at any given location in the nozzle
ρo = gas density in chamber **30**
ρ = gas density at any given location in the nozzle
A = area at any given location in the nozzle
A* = area at Mach Number unity, i.e., area at restriction or throat of the converging-diverging nozzle since $M_{throat}=1$
w = mass flow of gas
k = ratio of specific heats (specific heat of gas at constant pressure divided by specific heat at constant volume)
M = Mach Number.

By substituting any number equal to or greater than one for the Mach Number M in the above equations, representing gas flow greater than Mach 1 (supersonic gas flow), the relationships of pressures, temperatures, densities, and areas as noted may be determined so that suitable nozzle configurations may be made. The relationship given in equation (4) above is most useful, since changes of fluid properties in isentropic flow are brought about through changes in cross-sectional area.

Equation (4) may be employed to determine the area at the nozzle exit for any given nozzle throat area and desired Mach Number at the nozzle exit.

For a complete discussion of the known principles of supersonic fluid flows, see Ascher H. Shapiro, *The Dynamics and Thermodynamics of Compressible Fluid Flow* (Vol. 1, 1953), especially Chapter **4**.

Although the figures show a downward flow of molten metal to the atomization zone, a horizontal flow (not shown) is possible. The atomizing gas would be directed generally horizontally, rather than vertically. The trajectory of the atomized particles would be horizontal and then vertical. With such an arrangement the apparatus **10** would not be as high as the described apparatus which directs the inert gas generally downwardly.

One important feature of the invention is that before and/or after the molten metal has been atomized into particles within the atomizing zone **16***a* of the atomizing chamber **16**, additional matter is injected into the atomizing chamber **16** in order to cause the particles to agglomerate into particles of irregular and varied shape and to cool the metal particles to prevent them from forming a sintered mass. The additional injected matter may take the form of, e.g., an inert gas such as nitrogen gas preferably of 99.995 percent purity or fine particles of powder produced from atomization, preferably by recirculating fine particles separated from the main mass of atomized particles. This additional matter is injected into the atomizing chamber **16** and directed into the flow of metal particles through a series of secondary nozzles generically designated **22** in the drawings. It is possible to utilize both secondary inert gas and recirculated fine powder, as shown in the representative apparatus **10** of FIG. **1**. Employing the secondary injected matter produces an atomized powder which agglomerates and which forms a mass friable enough to be ground into particles of irregular shape but which does not form a tightly sintered mass in the atomizing chamber **16** nor contain massive oxide coatings or inclusions.

Inert gas, e.g., nitrogen gas, injected into the flow of metal particles at a pressure of about 30 pounds per square inch has been found effective to agglomerate the particles so that they are of the optimum shape for compacting into metal plate, sheet or strip. The inert gas may be injected into the atomizing chamber **16** through a valve **36** and a plurality of secondary nozzles **22***a* penetrating into the interior of the chamber **16** and directed substantially transverse to the flow of metal particles, as shown in FIG. **1**. Inert gas may also be directed substantially upwardly and against the flow of metal particles through nozzles **22***b* (as shown in FIG. **8**), in which case the gas also redirects any fine material dissipating from the flow of metal particles or accumulating in the collecting zone **16***b* back toward the flowing stream. Most of the agglomeration caused by the inert gas occurs with coarse particles rather than with fine particles, since the latter cool more rapidly by virtue of a higher surface area per unit mass and hence are not subject to agglomeration. Atomized powder injected with additional inert gas has been found to be of a lower density and to flow at a slower rate than powder produced without the injection of additional inert gas. Thus the powder so produced is particularly suitable for further processing.

Injection of fine particles of powder into the stream of metal particles results in more agglomeration of the latter as well as helps to cool the particles, and hence reduces the amount of tightly sintered material formed at the bottom of the atomizing chamber **16**, increasing the yield of metal powder. Moreover a produuct compacted from agglomerated particles has high strength. This fine powder is advantageously obtained by collecting the particles which leave the chamber **16** through an inert gas exhaust vent **37** near the atomizing zone **16***a* of the chamber **16**, the particles being recirculated at ambient temperature.

As shown in FIG. **1**, the additional powder, which may be contained in a plurality of hoppers **38**, may be injected into the stream of metal particles by means of a plurality of secondary nozzles **22***c* coupled to the hoppers **38** and penetrating into the atomizing chamber **16**, oriented so that the powder flow intersects the stream of metal particles at a point beneath the atomizing zone. Impellers **40** may be employed to direct the injected fine material into the stream of particles in a substantially transverse direction. The preferred amount of recirculated fine powder is about 400–600 pounds for a 500 pound charge of molten metal, or a ratio of about 1 part of fines to 1 part of molten metal, but a ratio of about 1.3 produces the lowest amount of sintered mass or cake. The embodiment of the apparatus shown in FIG. **1** produces few large particles which are ungrindable into usable powder. But the injection of fine particles of powder results in a more coarse product than when no secondary matter is injected. When no secondary powder is injected, only an ungrindable sintered mass and loose atomized powder are produced, whereas the addition of the injected matter results in a grindable sintered mass or cake which when ground may be combined with the loose powder to effect a high yield. The coarse product also contains a higher percentage of agglomerates than a fine product, thus resulting in a more usable product.

As shown in FIGS. **5–7**, other apparatus may also be employed to introduce fine particles of powder into the stream of molten metal and/or metal particles. In the device illustrated in FIG. **5**, fine particles of powder contained in the hopper **38** are discharged into streams of inert gas, e.g., nitrogen gas, which enter the chamber **16** at a point beneath the atomizing zone **16***a* thorugh nozzles **22***d* directed substantially transverse to the flow of metal particles. With this arrangement, the fine powder is fed by gravity into the stream of inert gas so that both forms of secondary matter may be injected into the produced particles simultaneously.

Fine particles of powder contained within the hoppers **38** may also be injected directly into the stream of molten metal by means of a plurality of nozzles **22***e* before the metal has been atomized into particles in the atomizing zone **16***a* (as shown in FIG. **6**). This method produces the highest percentage of coarse particles, especially when the rate of flow of fine particles is increased. The fine particles of powder cause the metal particles to agglomerate into particles of irregular and varied shape and to cool the particles to prevent them from forming a sintered mass whether the fine particles are introduced prior to atomization (FIG. **6**) or subsequent thereto (FIGS. **1** and **5**). With the embodiment shown in FIG. **6**, however, no supplementary nitrogen jets or impellers **40** are needed to direct the fine particles into the flowing stream, the gravity feed from the hoppers **38** being sufficient. Injection of fine particles of powder directly into the stream of molten metal insures that the secondary matter is thoroughly mixed with the metal.

It is further contemplated to inject fine particles of powder into the flow of molten metal and metal particles both prior to and subsequent to the atomization operation. As shown in FIG. **7**, a plurality of hoppers **38** similar to the hoppers utilized in the above-mentioned embodiments discharge fine particles of powder toward the atomizing zone **16***a* of the chamber **16.** Upon exiting each hopper **38,** the fine particles are divided into two streams, one of which is directed by means of nozzle **22***f* toward the flow of molten metal before it has been atomized and the other of which is directed by means of nozzle **22***g* toward the flow of metal particles after atomization. Both streams of secondary particles are injected in the direction of flow of the molten metal and metal particle stream, so that the injection may be accomplished merely by a gravity feed, without the utilization of secondary nitrogen jets or impellers **40.** This method results in a less coarse powder than the method employing the apparatus of FIG. **6.** However, the particle size increases as more fine particles of powder are injected before atomization as opposed to post atomization, i.e., as the method more closely approximates the device of FIG. **6.** Greater control over particle size and extent of agglomeration is offered by the FIG. **7** apparatus.

It is apparent that secondary matter may be injected at a plurality of locations, both before and after atomization, as well as in the zone of atomization. ITt should also be noted that the zone of atomization might include a series of discrete gas sources spaced one from another in the general direction of movement of the stream of molten level so as to provide atomization at a number of levels. For example, in the apparatus of FIG. **7,** atomizing gas could be directed not only from the nozzle **20** but also from similar nozzle structures located as the nozzles **22***f* and **22***g*. Such a plurality of levels of atomization might be useful for relatively large streams of molten metal, although the problem of nozzle clogging might be encountered.

Generally, fine particles of powder less than 65 mesh in size have been found most suitable for secondary injection. This fraction of the product contains a relatively high percentage of spherical particles which, if subsequently compacted into steel plate, strip, or the like, results in an intermediate product of insufficient strength for further processing. In many test runs it was found that although the use of the −65 mesh powder was effective to produce agglomerates, the amount of fines needed exceeded the amount generated. To maintain a material balance in the system, it became necessary to recycle as well a portion of the −8+65 mesh product. Since this more coarse material does not require further agglomeration, it was injected solely to minimize the tendency of the atomized particles to form a sintered mass or cake during the collecting and cooling stages. Use of the more coarse product was particularly effective with the embodiment shown in FIG. **7.**

Subsequent to the injection of secondary material into the stream of metal particles, the particles pass into the collecting zone **16***b* of the chamber **16.** It has been found that the particles which settle in the relative center of the collecting zone **16***b* form a partially sintered product, which when ground produces particles having a desired irregular shape. Thus the particles taken from the relative center of the collecting zone **16***b* are especially suited for compaction into metal plate, sheet, or strip.

In FIG. **1**, the collecting zone **16***b* of the atomizing chamber **16** is shown associated with a rotary cooler **42.** The rotary cooler **42** may be utilized to transport the powder product to a station for further processing. As an alternative means of transporting the metal powder from the collecting zone **16***b* to the next processing station, an enclosed container **44** may be provided, as shown in FIG. **9.** A container **44** defining a conveying device is presently preferable as it collects the produced powder before the particles become sintered or caked, which can readily occur as the powder enters the collecting zone **16***b* at a temperature of about 970° F. Such a device **44** includes hopper plates **46** located at the base of the collecting zone **16***b* and adapted to discharge the produced metal powder onto a moving conveyor belt **48** enclosed by a housing **50.** The hopper plates **46** are inclined inwardly so that metal powder will more readily flow onto the conveyor belt **48.** A plurality of nozzles **52** may be provided for the injection of further inert gas, e.g., nitrogen gas, or fine particles of powder onto the hopper plates **46** to prevent the metal particles from adhering to the hopper plates and hindering the flow of material onto the conveyor belt **48.** A rotary cooler **42** or a conveyor belt **48** is an integral part of the invention as it provides in conjunction with the secondary injected matter a means for dissipating heat from the produced particles so that the particles do not form a sintered mass or cake.

EXAMPLE 1

In one operation of the process using auxiliary fine particles of metal powder and a device as shown in the upper portion of FIG. **1**, an AKDQ steel was used to produce a metal powder, the steel having the following characteristics (given as percentages of the melt by weight):

Carbon	0.06 – 0.08
Aluminum	0.03 – 0.08 (0.05 desired)
Manganese	0.28 – 0.32
Oxygen	0.012 – 0.030
Nitrogen	0.008 Max.
Sulfur	0.02 Max.
Phosphorus	0.02 Max
Silicon	0.01 Max.

The atomizing chamber **16** (having a volume of approximately 315 cubic feet and formed from 12 feet of culvert sections 5 feet in diameter welded to a rectangular sheet container 5 feet × 4 feet × 3 feet) was preliminary purged with nitrogen gas at the rate of 190 cubic feet per minute for about 30 minutes prior to atomization. During atomization the chamber **16** was maintained at a gas temperature of 250° to 300° F and a pressure of 1.1 inches of water. 500 pounds of molten steel were discharged from the tundish **12** at a temperature of 3,110° F and at a rate of 180 pounds per minute through a nine-sixteenths-inch diameter feeding nozzle in the tundish. A 4-inch inside diameter nozzle **20** comprising a plurality of gas jets **28** of the converging-diverging type (FIG. **2**) with an included gas angle of 35° was employed to atomize the stream of molten steel into particles. The atomizing inert gas was nitrogen gas of 99.995 percent purity and was directed toward the stream at the rate of 1,500 cubic feet per minute at a pressure of 130 pounds per square inch. In addition, secondary fine particles of metal powder (−65 mesh) were injected into the stream of particles below the atomization zone by impellers such as impellers **40** in the amount of about 1.3 pounds of fine particles per pound of molten metal and at a rate of about 124 lbs. per min.

The product collected from the process was classified into three distinct categories: atomized fines, atomized powder, and atomized sinter. Atomized fines exited the atomizing chamber **16** through the inert gas exhaust vent **37.** These fines made up 6.7 percent (by weight) of the product and contained 0.035 percent oxygen and 0.084 percent carbon. The cumulative sieve analysis of the fines was as follows (in percent by weight):

+28 mesh	0
+35 mesh	0.28
+48 mesh	2.90
+65 mesh	12.97
+100 mesh	32.25
+150 mesh	57.42
+325 mesh	91.00

The bulk of the atomized steel (48.8 percent by weight) was atomized directly into powder (excluding fines). 1.9 percent (by weight) of this powder was +8 mesh in size and 98.1 percent was− 8 mesh, that size being arbitrarily chosen to indicate ungrindable sinter. The −8 mesh portion had a density of 3.72 grams per cubic centimeter and the following chemical composition (in percent by weight):

Carbon	0.070
Aluminum	0.03
Manganese	0.26
Silicon	0.010
Phosphorus	0.003
Sulfur	0.017
Nitrogen	0.006
Oxygen	0.018

Of the 98.1 percent (by weight) of the powder which was less than 8 mesh, the cumulative sieve analysis (in percent by weight) was as follows:

+14 mesh	5.44
+20 mesh	13.25
+28 mesh	23.94
+35 mesh	34.73
+48 mesh	43.86
+65 mesh	53.69
+100 mesh	65.13
+150 mesh	79.66
+325 mesh	96.88

Much of the medium-sized powder formed was of irregular shape and coarse in nature. Ten pound samples of produced powder flowed for about 33 to 42 seconds through a 0.5 inch diameter orifice. The powder was found to be quite suitable for optimum compacting into steel plate.

Finally, the atomized sinter constituted 44.5 percent (by weight) of the product. Much of the sintered product was eliminated and that which remained was readily ground into useful powder, producing a total of 93.3 percent (by weight) powder.

EXAMPLE 2

In another operation of the process using the apparatus such as shown in FIG. **6,** particles of AKDQ steel powder of the same chemistry of the steel of Example **1** were agglomerated and cooled. The atomizing chamber **16** was of the same dimensions and was initially prepared in the same manner as explained in Example **1.** The molten steel was introduced into the atomizing chamber **16** at about the same parameters as disclosed in Example **1.** A 4-inch inside diameter nozzle **20** comprising a plurality of gas jets **28** of the converging-diverging type (FIG. **2**) with an included gas angle of 30° was employed to atomize the stream of molten metal into particles. The atomizing inert gas was of the same type and flowed at about the same rate and pressure as the gas used in the process of Example **1.** Additional fine particles of metal powder (−65 mesh) were injected directly into the stream of molten metal by nozzles **22*e*** prior to atomization in the amount of about 0.85 lbs. of fine particles per pound of molten metal and at a rate of about 144 pounds per minute.

Again the product collected from the process was classified into atomized fines, atomized powder and atomized sinter. The cumulative sieve analysis of the fines was approximately the same as for the fines of Example 1.

The cumulative sieve analysis (in percent by weight) of the produced usable powder (including reground sinter) was as follows:

+10 mesh	0.51
+14 mesh	39.57
+20 mesh	61.43
+28 mesh	76.07
+35 mesh	84.96
+48 mesh	90.79
+65 mesh	95.02
+100 mesh	98.78
+150 mesh	99.42
+325 mesh	99.84

This method produced a high percentage of coarse particles of irregular shape. Ten pound samples of produced powder flowed for about 54 to 67 seconds through a 0.5 inch diameter orifice. Again the powder was found to be suitable for compacting into steel plate, strip, or the like.

Examples 1 and 2 show that particle injection prior to atomization (Example 2) produces a coarser product than injection following atomization (Example 1).

EXAMPLE 3

Further operations of the process were conducted using apparatus such as shown in FIG. **7.** Again an AKDQ steel having the characteristics of the first example was employed and the atomizing chamber **16** was of the same dimensions and was preliminarily prepared in the same manner. Moreover, 500 pounds of molten steel were again discharged from the tundish **12** at a temperature of about 3,100° F and at a rate of about156 to 170 lbs. per minute through a nine-sixteenths-inch diameter feeding nozzle in the tundish. A 3-inch inside diameter nozzle **20** comprising a plurality of gas jets **28** of the converging-diverging type (FIG. **2**) with an included gas angle of 30° was employed to atomize the stream of molten steel into particles. The atomizing inert gas had about the same parameters as the inert gas used in the operation of Example 1. Secondary fine particles of metal powder were injected into both the stream of molten metal as by nozzles **22*f*** (injected particles −65 mesh) and the stream of metal particles as by nozzles **22*g*** (injected particles −65 mesh).

Again the fine particles produced were of approximately the same size as the particles produced in Example 1. The usable powder produced by this process varied in particle size according to the ratio of powder injected above the atomization zone to powder injected below the atomization zone. Table 1 indicates the cumulative sieve analysis (in percent by weight) for various ratios, amount of powder injected, and particle flow rates as follows:

Table 1

Ratio	0.20	0.30	0.65	1.00	1.36
Total Powder Injected (lb.)	515	560	610	400	465
Powder Flow Rate (lb. per min.)	212	208	228	144	170
Particle Size (mesh)	Cumulative Sieve Analysis			(% by weight)	
−8+10	0.13	0.10	0.11	0.27	1.39
+14	7.32	23.33	28.38	37.39	46.97
+20	18.48	43.31	48.36	59.39	66.11
+28	33.73	59.43	61.83	74.66	77.26
+35	49.73	72.13	71.90	85.49	84.52
30 48	66.16	82.12	80.03	91.93	89.60
+65	83.08	90.12	87.03	96.35	93.73
+100	96.51	97.29	96.09	99.22	98.35
+150	98.41	98.64	98.61	99.62	99.20
+325	99.31	99.41	99.61	99.99	99.73

Thus it can be seen that as more particles of powder are injected into the molten metal prior to atomization as opposed to injection subsequent to atomization, the size of the produced particles increases, resulting in desirable intermediate-size powder. Ten pound samples of usable powder flowed for about 38 to 61 seconds through a 0.5 inch diameter orifice. Only about 23 pounds of +8 mesh material was produced using apparatus such as shown in FIG. 7 and after regrinding, these particles were added to the intermediate particles and included in the cumulative sieve analysis of Table 1. Once again, the process resulted in metal powder suitable for optimum compacting into metal plate or strip. The data in Table 1 shows the control over the constituency of produced powder possible through varying the ratio of injected particles prior to and following atomization.

EXAMPLE 4

Using apparatus such as shown in FIG. 5 for introducing both secondary inert gas and fine particles of powder into the stream of produced metal particles, agglomeration and cooling of the particles resulted. An AKDQ steel of about the same nature, in about the same amount, and flowing at about the same rate as that used in Example 1 was introduced into the atomizing chamber (which was prepared in the same manner indicated in Example 1). The steel was atomized using a converging-diverging type nozzle **20** (FIG. 2), the atomizing inert gas having about the aforementioned parameters of the first example. Additional fine particles of metal powder (−65 mesh) were injected into the stream of metal particles after atomization in the amount of about 0.4 lbs. of fine particles per pound of molten metal at a rate of about 67 lbs. per minute. The injection was accomplished by gravity feeding the fine particles into a stream of nitrogen gas flowing at a pressure of about 40 lbs. per sq. in. and directed toward the stream of produced particles by nozzles **22*d***.

The fine particles of powder produced were similar in size to the particles produced in the above examples. The cumulative sieve analysis (in percent by weight) of the produced usuable powder (including reground sinter) was as follows:

+14 mesh	0.0
+20 mesh	3.78
+28 mesh	12.75
+35 mesh	29.36
+48 mesh	51.01
+65 mesh	79.21
+100 mesh	97.51
+150 mesh	98.65
+325 mesh	99.39

A desirable powder of intermediate size was thus produced, the metal powder being appropriate for further processing.

EXAMPLE 5

Auxiliary nitrogen gas was also employed to agglomerate and cool particles of metal powder produced from an AKDQ steel (the steel having the characteristics of the first example) using apparatus such as shown in FIG. **8.** The atomizing chamber **16** was of the same dimensions and was initially prepared in the same manner as in the process using the device of Example 1. 500 lbs. of molten steel were discharged from the tundish **12** at a temperature of about 3,105° F and at a rate of about 172 lbs. per minute through a nine-sixteenths-inch diameter feeding nozzle in the tundish. A 3-inch inside diameter nozzle **20** of the converging-diverging type (FIG. **4**) having a 0.040 inch annulus and defining an included gas angle of 10° was employed to atomize the stream of molten metal into particles. Nitrogen gas of 99.995 percent purity was directed toward the stream at the rate of 1,100 cu. ft. per minute at a pressure of 115 lbs. per sq. in. to atomize the stream. Additionally, nitrogen gas of 99.995 percent purity was injected into the stream of particles by secondary nozzles such as **22*b*** at the rate of 240 cu. ft. per minute at a pressure of 20 lbs. per sq. in.

The product of the process was again classified into atomized fines, atomized powder, and atomized sinter. The fines comprised 8.6 percent (by weight) of the product and were of the same content and size as the fines of Example 1.

Initially, about 51.7 percent (by weight) of the product completed the process as powder. 0.7 percent (by weight) of the powder was +14 mesh in size and 99.3 percent was −14 mesh. The −14 mesh portion had a density of 4.27 grams per cubic centimeter and the following chemical composition (in percent by weight):

Carbon	0.084
Aluminum	0.066
Manganese	0.32
Silicon	0.011
Phosphorus	0.008
Sulfur	0.018
Nitrogen	0.012
Oxygen	0.021

Of the 99.3 percent (by weight) of the powder which was −14 mesh, the cumulative sieve analysis (in percent by weight) was as follows:

+20 mesh	1.12
+28 mesh	4.84
+35 mesh	17.32
30 48 mesh	35.30
+65 mesh	58.78
+100 mesh	78.06
+150 mesh	90.43
+325 mesh	98.71

The majority of the intermediate sized powder was of irregular shape and coarse in nature. A 25 cubic centimeter sample of produced powder flowed for 23.3 seconds through a 0.2 inch diameter orifice. The powder was found to be suitable for optimum compacting into steel plate although it was not highly agglomerated.

The atomized sinter constituted 39.7 percent (by weight) of the product. Of the sintered proudct the bulk could be readily ground into usable powder so that the total amount of powder was 91.4 percent (by weight). Only about 1 percent of the molten metal was eventually found to be unusable.

In order to test the compactability of the atomized powder, samples of powder from various runs were formed into briquettes at a pressure of about 80,000 psi in a 1 inch diameter die, that pressure being found necessary to keep the briquettes of lesser agglomerated powder intact. Upon applying a load to the briquettes, it was found that those containing a higher percentage of agglomerates withstood a higher compression load without rupture, as the agglomerates allow for particle interlock upon compression. The amount of compression load endured was also found to be a function of the length of time of powder flow and the apparent density of the powder.

Thus, the present invention provides apparatus and process for atomizing molten metal into a metal powder possessing optimum properties for compaction into metal plate, sheet, or strip, for example. The invention allows a metal powder to be produced in agglomerated form, which results in stronger particle-to-particle bonds in the compacted product, but without particles containing oxide coatings and without a sintered mass being formed at the base of the atomizing chamber which could not be readily ground into particles of desired size and shape.

We claim:

1. Apparatus for the production of metal powder, comprising:

a. tundish means adapted to provide a smooth flowing stream of molten metal;

b. an atomizing chamber into which said stream of molten metal flows, containing an atomizing zone and a collecting zone;

c. atomizing means for producing particles of metal from said molten metal stream;

d. injecting means for directing particles of the same material as the molten metal against the metal in said atomizing chamber to cause the atomization-produced particles of metal to agglomerate into irregular shapes and to cool them; and

e. collecting means for receiving said metal particles in said collecting zone.

2. Apparatus as defined in claim 1, wherein said injecting means includes impeller means for directing said particles of material against the metal in said atomizing chamber.

3. Apparatus as defined in claim 1, wherein said injecting means includes means for entraining said particles of material in a gas which is directed against the metal in said atomizing chamber.

4. Apparatus as defined in claim 1, wherein said injecting means injects said particles of material against said stream of molten metal.

5. Apparatus as defined in claim 1, wherein said injecting means injects said particles of material against the atomization-produced particles of metal.

6. Apparatus as defined in claim 1, wherein said injecting means injects said particles of material concurrently against said stream of molten metal and the atmoization-produced particles of metal.

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