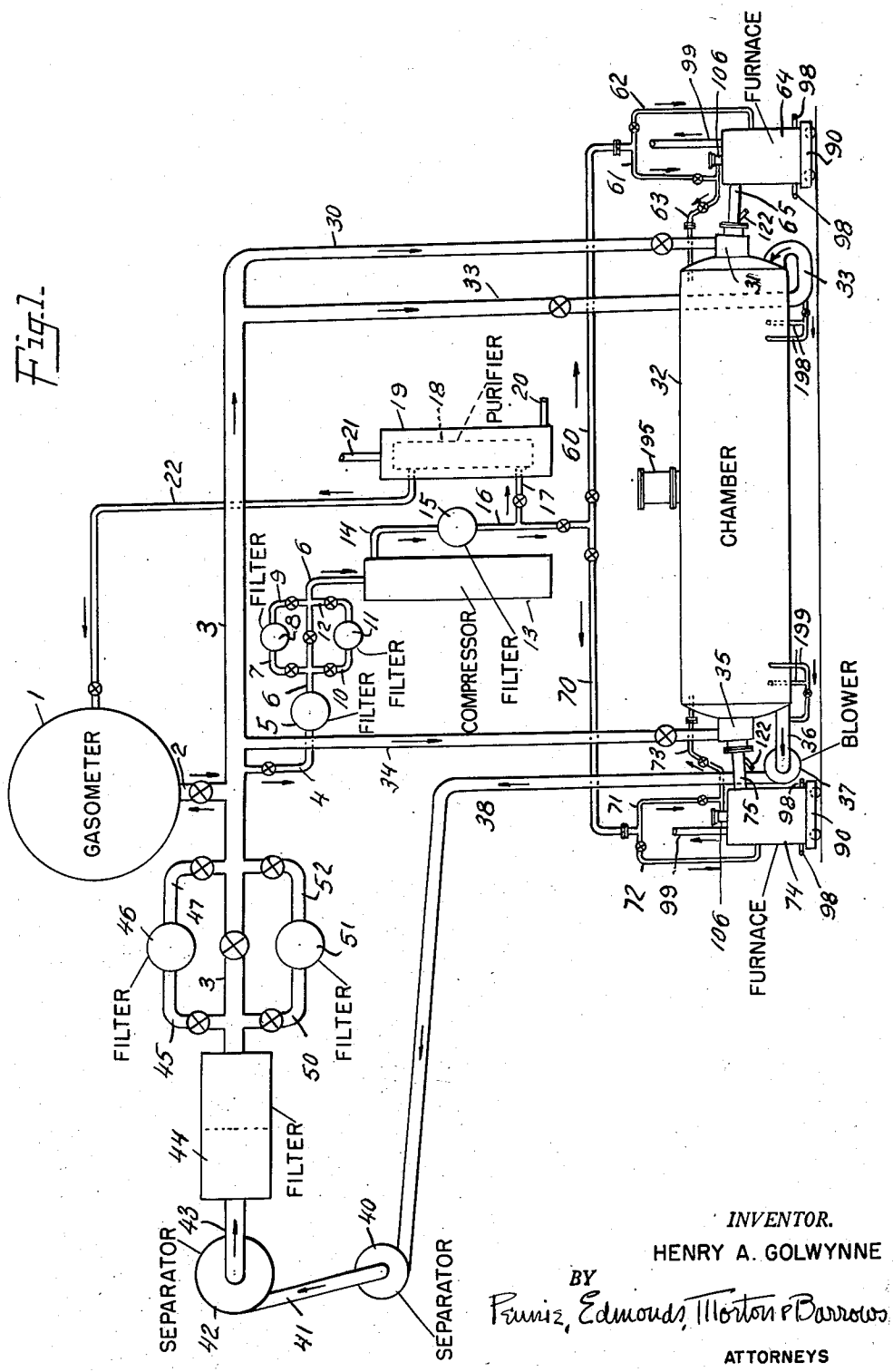
May 19, 1953 H. A. GOLWYNNE 2,638,630

PRODUCTION OF METAL POWDER

Filed Sept. 29, 1949 5 Sheets-Sheet 1

Fig. 1.



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PRODUCTION OF METAL POWDER

Filed Sept. 29, 1949 5 Sheets-Sheet 2

Fig. 2.

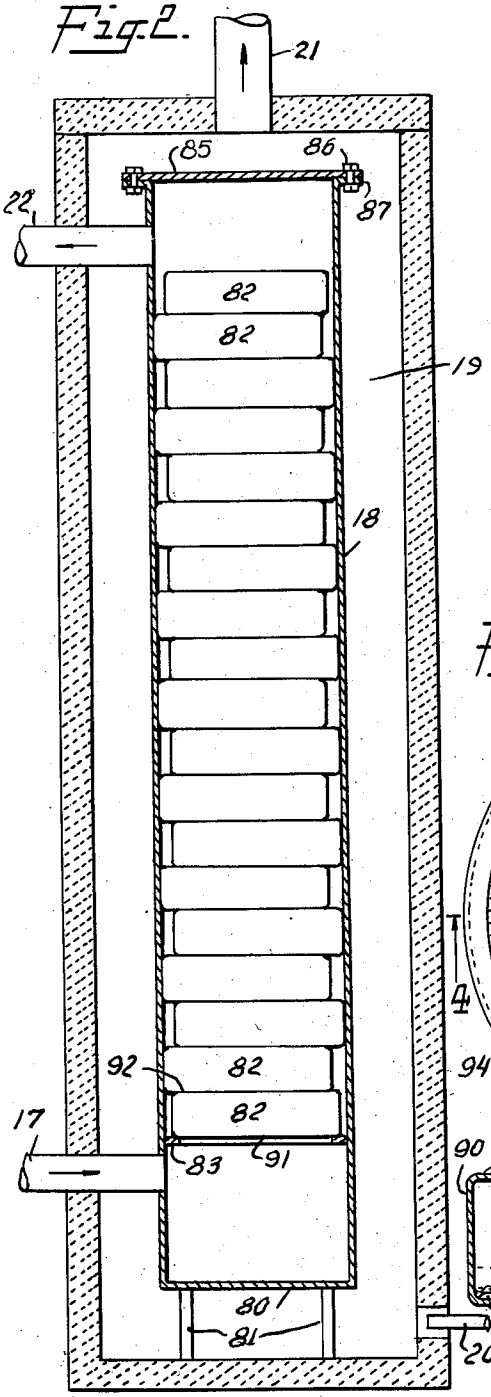


Fig. 9.

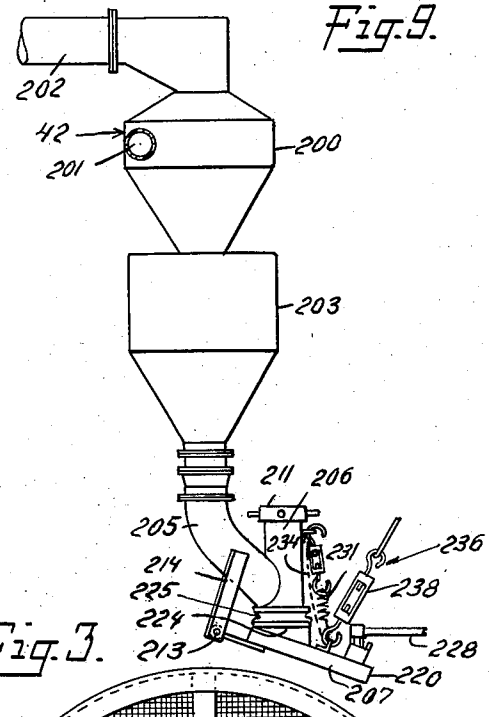


Fig. 3.

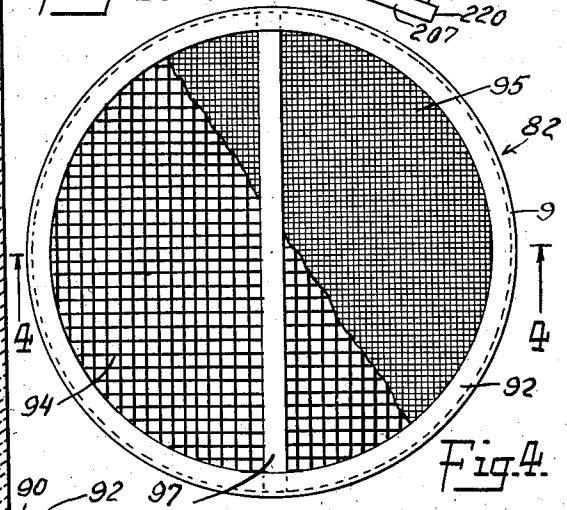
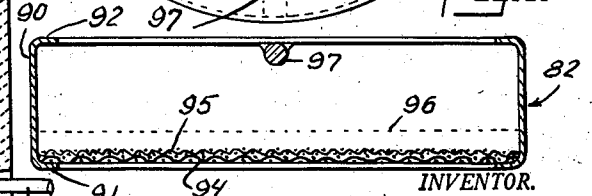


Fig. 4.



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PRODUCTION OF METAL POWDER

Filed Sept. 29, 1949 5 Sheets-Sheet 3

Fig. 5.

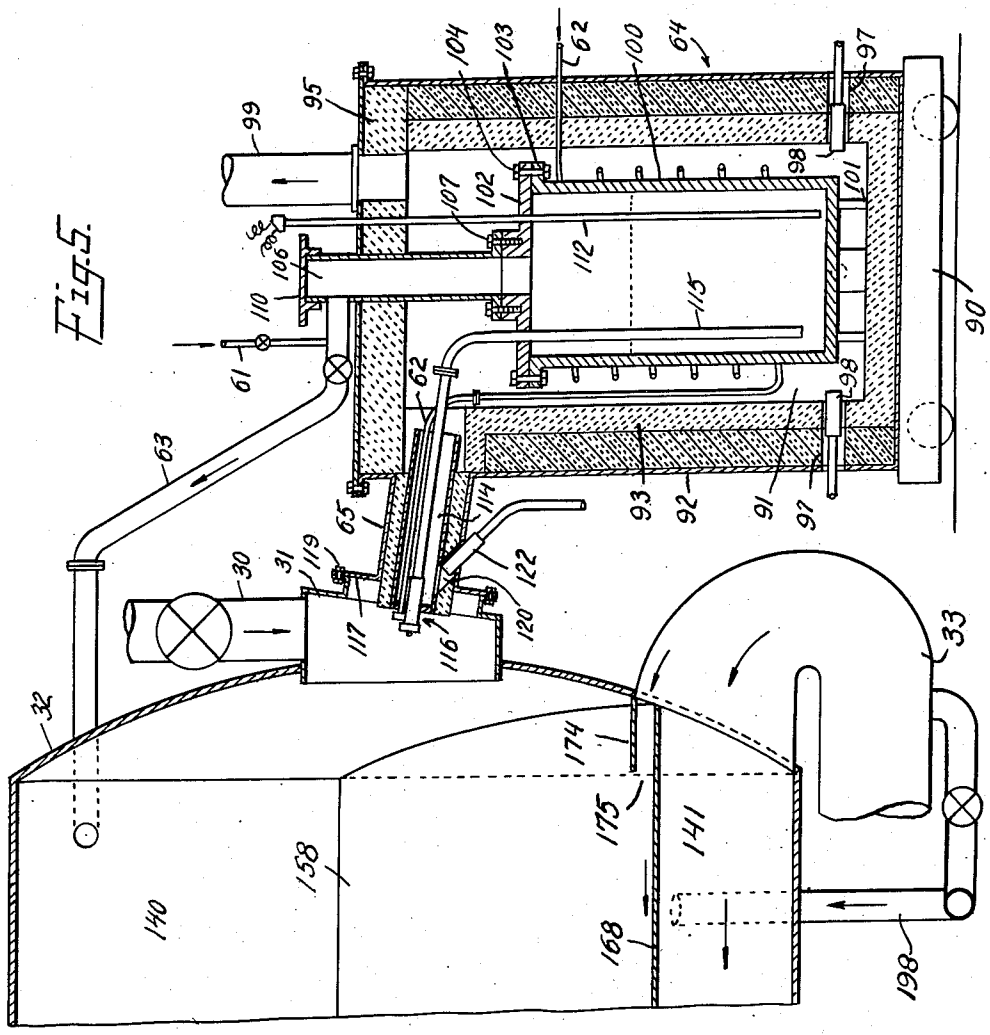
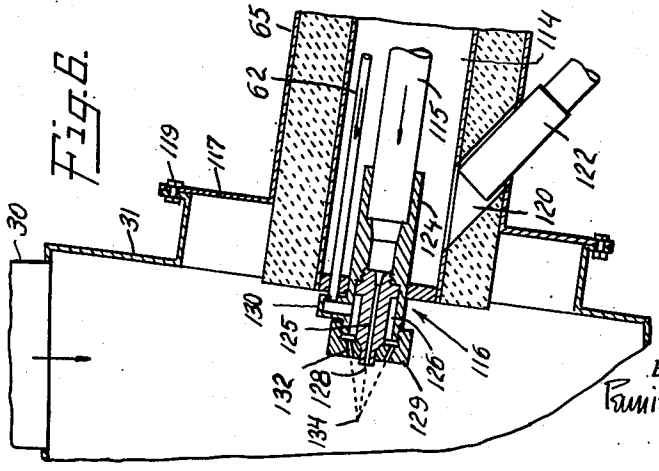


Fig. 6.



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PRODUCTION OF METAL POWDER

Filed Sept. 29, 1949 5 Sheets-Sheet 4

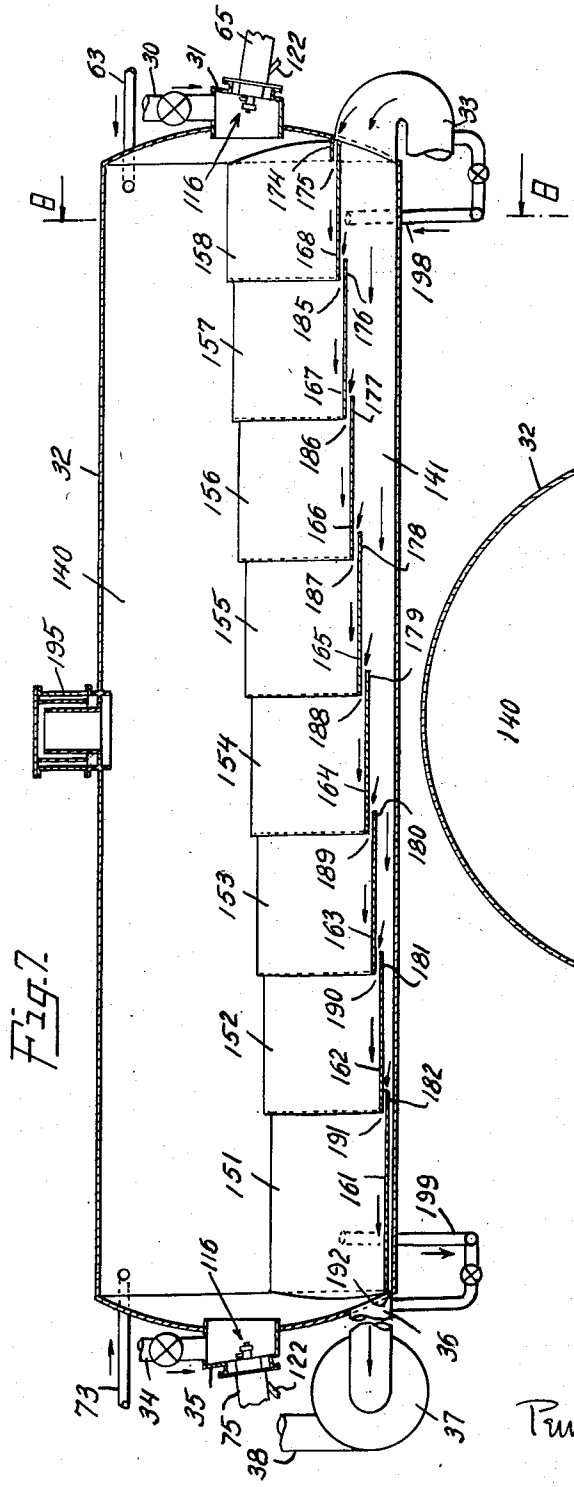


Fig. 7.

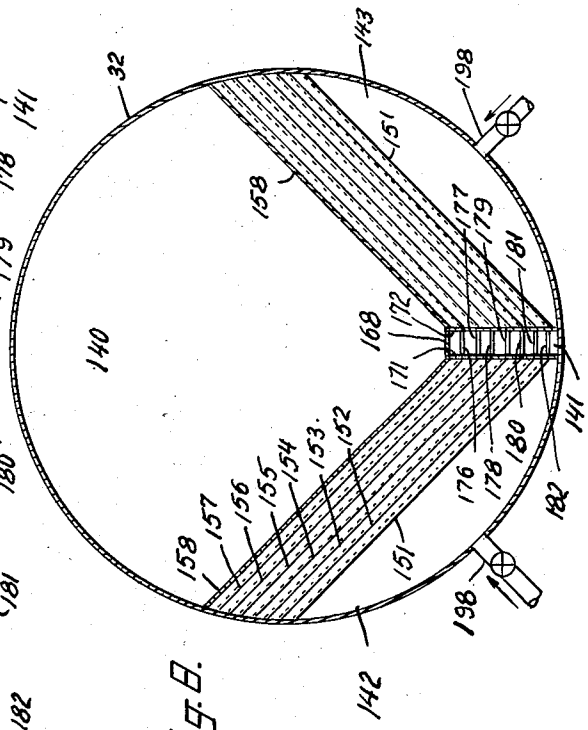


Fig. 8.

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PRODUCTION OF METAL POWDER

Filed Sept. 29, 1949 5 Sheets-Sheet 5

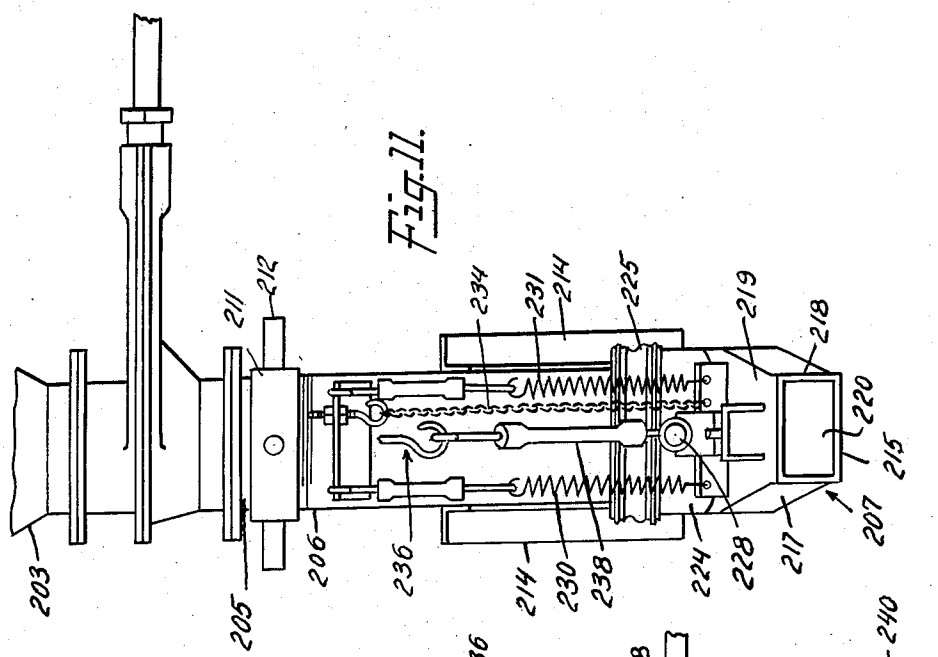


Fig. 11.

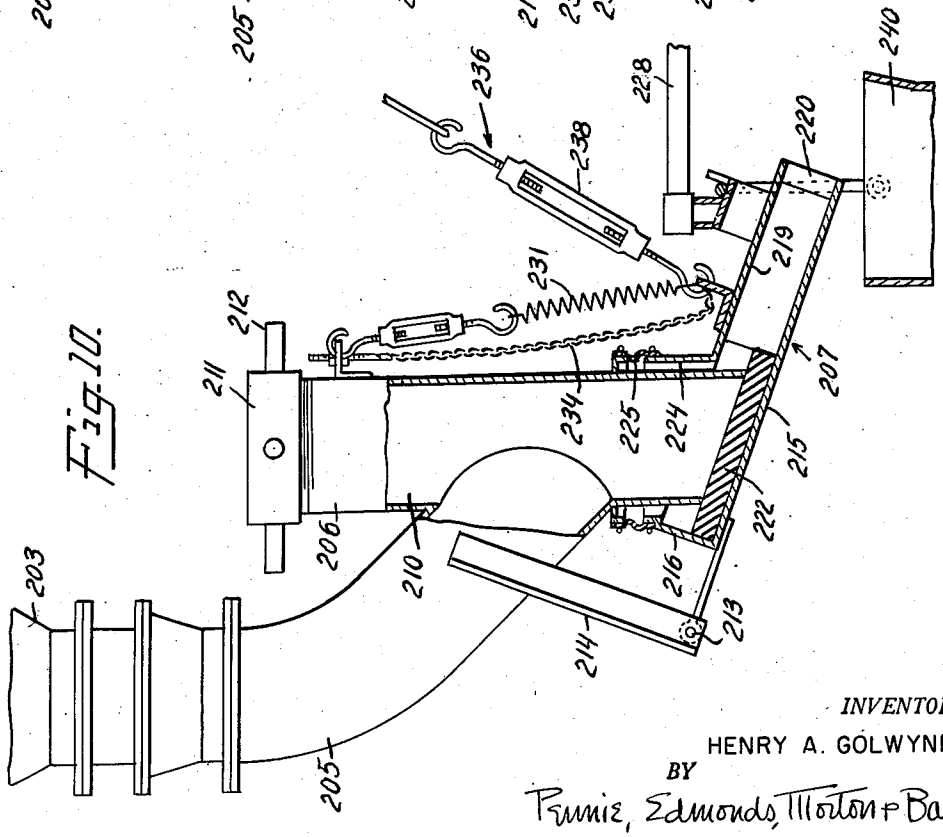


Fig. 10.

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Patented May 19, 1953 2,638,630

UNITED STATES PATENT OFFICE

2,638,630

PRODUCTION OF METAL POWDER

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Application September 29, 1949, Serial No. 118,621

20 Claims. (Cl. 18—47.3)

1

This invention relates to the production of metal powders and has for its object improvements in the method of producing metal powder.

Among the methods of producing metal powder is that of atomizing molten metal with a blast of gas, freezing the atomized metal into finely divided solid particles, and recovering the resulting metal powder. Most metals have a marked affinity for oxygen and nitrogen at elevated temperatures. This makes it difficult to produce metal powder from molten metal by atomization with a gas because the oxides and nitrides formed by the reaction of oxygen and nitrogen in the gas with the atomized metal tend to form at or near and to contact and then to adhere to the highly heated atomizing nozzle, thus clogging or otherwise impairing its usefulness. Such nozzles usually have a central outlet or discharge opening through which the molten metal is forced and usually a plurality of ports through which streams of the gas are forced against the molten metal discharged from the outlet. Although a great deal of the oxide and nitride finds its way into the metal powder produced, appreciable amounts contact the hot nozzle and adhere thereto as an accretion or incrustation. This builds up around the ports, particularly, and even the outlet, eventually, so that they become smaller and smaller and clogged eventually. As this takes place the size of the molten metal and gas streams is gradually reduced, thus altering radically the conditions under which the powder is formed. This affects the particle size of the product as well as the efficiency of the operation. The nozzles must be removed, repaired or replaced frequently.

In the earlier attempts to produce metal powder by atomization, the gas employed for atomization and in the atomizing chamber was air or steam. Air is highly oxidizing and nitriding and steam mildly oxidizing. They have been discarded in favor of so-called inert or non-reacting gases. As we shall see inert gases available commercially in quantities are not truly inert, and even if truly inert other oxidizing and nitriding influences are inherent in the operation. In any event various proposals have been advanced to atomize molten metal with and in the presence of such inert or non-reactive atmospheres. For example, a trickle or stream of molten metal is blasted in a chamber filled with inert gas; the atomized metal is frozen into finely divided solid particles; and the resulting metal powder permitted to settle at the bottom of the chamber from which it is removed periodically. Another proposal is to spray molten magnesium into a chamber filled with cold nitrogen gas deemed to be comparatively inert to the magnesium. Magnesium, however, has a high affinity for nitrogen, when either or both is heated, and it is impossible to spray molten magnesium, which is necessarily highly heated, in such a manner without providing a zone, even though restricted in extent, conducive to the formation of an objectionable amount of magnesium nitride. As before the powder is removed periodically from the bottom of the chamber.

2

Investigators in this field have been greatly troubled with the problem of producing a sufficiently inert or non-reactive atmosphere in which to atomize the molten metal. As manufactured, the gas itself, for example helium, argon, etc., is not entirely inert because it usually contains a certain amount of impurities such as oxygen and nitrogen. The inert gas must be conducted into the chamber in which the molten metal is to be atomized and the powder formed. Since the chamber is initially filled with air, the air must be flushed out, or otherwise be replaced, by the inert gas. Since gases mix readily, the inert gas and air promptly intermix. In spite of careful precautions a residuum of air remains, and since the air is rich in oxygen and nitrogen, the atmosphere as a whole in the chamber is contaminated to that extent. The atomized metal reacts readily with the oxygen and nitrogen. Oxide and nitride incrustations then form on the nozzle used to atomize the molten metal. The discharge ports of the nozzle are soon clogged and the operation must be stopped to clean or replace the nozzle.

My investigations have led to the discovery that difficulties and disadvantages of the type discussed may for the most part readily be overcome. With certain important improvements in the method of producing metal powder by atomizing molten metal with a blast of inert gas, metal powders may be produced steadily over prolonged periods. Metal powders substantially uniform in quality and in particle size may be obtained. The operation may be so conducted as to obtain metal powders graded generally as to particle size.

In accordance with one aspect of the invention, a confined circulatory system—including molten metal atomizing, metal powder forming and powder separating zones—is filled initially with inert gas under pressure higher than atmospheric to prevent ingress of outside air. The inert gas in the system is purified with respect to such impurities as oxygen and nitrogen, after which metal powder is produced therein.

Air initially present in the circulatory system is replaced for the most part with inert gas. The inert gas is introduced in the system at a high point and the undesired air containing oxygen and nitrogen is withdrawn at a low point. The oxygen and nitrogen in the inert gas and in the residue of air remaining with the inert gas in the system are then eliminated.

The purification operation is advantageously conducted by causing the oxygen and nitrogen impurities in the gas to react with heated metal particles to form the oxide and nitride of the metal. Metal powder of the kind to be produced, or previously produced, is preferably employed because it is at hand. The gas may be withdrawn temporarily from the system, purified, and then returned to the system; the entire operation being a circulatory or cyclical one until all of the gas in the system has been in contact with the heated metal for purification and has been returned to the system.

According to another aspect of the invention, the molten metal atomizing, metal powder forming and powder separating steps are conducted in the confined circulatory system filled with the purified inert gas as the gas is continuously circulated therethrough under pressure higher than atmospheric to prevent ingress of outside air. This is advantageously accomplished by blasting a fine stream of the molten metal circumferentially with a plurality of streams of heated inert gas in the metal powder forming zone filled with the circulating inert gas, the circulating gas being sufficiently low in temperature promptly to freeze the atomized metal into finely divided particles. The circulating inert gas with metal powder suspended therein is passed from the powder forming zone to a powder-gas separating zone; metal powder is separated from the inert gas; the inert gas remains in the system to be reused; and the metal powder so separated is withdrawn from the system.

To obtain the plurality of fine streams of inert gas to atomize the molten metal, it is advantageous to withdraw temporarily some of the inert gas from the system, place it under substantial positive pressure, and return it to the system by atomizing the molten metal. The gas so withdrawn from the system is preferably preheated by placing it in heat-interchange relationship with the body of molten metal to be atomized. The fine streams of heated inert gas are then forced under high pressure into the stream of molten metal thus breaking it up into a myriad of fine droplets which are then enveloped in the cooler inert gas circulating through the system and frozen into solid particles. If cool gas were used to atomize the molten metal, at least in the atomizers conventionally employed, molten metal would tend to freeze and adhere to the discharge tip.

Another highly advantageous expedient is to place the source or body of molten metal to be atomized under substantial pressure with the inert gas so that molten metal may be forced as a stream into the atomizing zone. To this end some of the inert gas is withdrawn from the system, conducted under substantial positive pressure to a confined space above the body of molten metal and used to force a stream of the molten metal to the atomizing zone.

In a presently preferred practice of the invention, the circulating inert gas with metal powder suspended therein is passed from the powder forming zone through a plurality of powder-gas separating zones, metal powder being separated from the inert gas in each zone and the metal powder so separated is withdrawn from the system at each zone.

The separation of the metal powder from the gas may be conducted in various ways. At the present time the gas with metal powder suspended therein is passed successively through a series of cyclones so that the separation of the powder may take place by dry precipitation. This has the advantage that powder of graded particle size may be selectively separated in each cyclone; for example, fine particles in the first cyclone, finer particles in the next cyclone, and so on. The residue of dust remaining in the gas leaving the last cyclone is advantageously thrown down by wet precipitation, such as by passing the gas through a bag filter wet with oil. As an additional precaution it is desirable to use a filter in the line going to the compressor.

As already indicated, improvements in the apparatus employed are necessary to obtain the advantages of the method. The apparatus for producing the metal powder comprises a confined circulatory system adapted to be filled with gas under pressure higher than atmospheric; a gasometer to hold surplus gas and to take care of expansion and contraction of the gas; a chamber for atomizing molten metal and chilling atomized metal into powder; a blower for circulating the gas around the system; and a separator for separating the powder from the gas.

An atomizer for the molten metal extends at its discharge end into the chamber. The atomizer in turn connects with the bottom portion of a melting furnace and means are provided for exerting pressure inside the furnace so that molten metal at the bottom may be forced through the atomizer into the chamber. This pressure is preferably obtained with a compressor connected on the inlet side with the system for the withdrawal of some gas and on the outlet side with the interior of the melting furnace to place a layer of gas over the body of molten metal under sufficient pressure to force molten metal through the atomizer. The compressor preferably connects on the inlet side with the system beyond the place or places where the powder is separated from the gas so that substantially powder-free gas may be passed by the compressor to the furnace.

Another advantageous arrangement is one adapted to use some of the gas in the system for atomizing the molten metal. To this end the compressor, as before, connects on the inlet side with the system for the withdrawal of some gas and on the outlet side with a conduit in heat-interchange relationship with the furnace for melting the metal to be atomized, the discharge end of the conduit connecting with the atomizer for the molten metal so that the molten metal discharged from the atomizer may be blasted with gas preheated by the furnace. Also, as just described above for the furnace, the compressor preferably connects on the inlet side with the system beyond the place or places where the powder is separated from the gas so that substantially powder-free gas may be passed by the compressor through the preheating conduit to the atomizer for atomizing the molten metal.

A combination of the features just described is highly desirable in practice. In other words, the compressor connects on its outlet side with the interior of the furnace as well as with the atomizer, so that molten metal in the furnace

may be placed under pressure by the gas and thus be forced from the furnace to and through the atomizer and so that the molten metal may be atomized by the gas from the compressor when it is discharged into the chamber. The compressor connects on the inlet side with the system beyond the place where the powder is separated from the gas so that substantially powder-free gas may be passed by the compressor to the furnace and to the atomizer.

The invention is adapted for the production of a variety of metal powders. The metal to be converted into powder may be essentially a primary metal or a combination of metals, such as alloys. As a practical matter one of the determining factors is, of course, the melting point of the metal. The higher the melting point, the more are the difficulties encountered; particularly as regards heating and wear and tear of the equipment. The invention is being currently practiced in the production of magnesium powder, and powders of alloys of magnesium, more particularly alloys of magnesium and aluminum.

While various gases inert to the metal to be converted to powder may be used, as a practical matter the choice simmers down to one readily available at a relatively low cost. For this reason helium is now being used. Other inert gases, such as argon could, of course, be employed.

The practice of the invention as described results in an unusually stable and efficient product. The metal powder may be stored without appearing to lose its effectiveness in use. In the case of magnesium, the powder remains highly effective after storage. This is not true of conventional magnesium powders such as are produced by grinding or attritional means. This may be due to the fact that such powders are produced in a relatively hot state due to the grinding or attritional friction, which causes the exposed surfaces of the particles to react with the surrounding gases and thus, for example, to take on a coating of oxide. On the other hand, powder of the present invention is produced in a relatively cool atmosphere due to the large volumes of cool circulating inert gas that envelop and chill the particles promptly on formation, and carry them away from the atomizing and powder forming zones. There is little or no opportunity for an oxide coating to form on the individual particles.

In addition the metal powder of the invention is highly activated thus making it very efficient in use. This appears to be due to the enormous amount of surface area offered by a body of the product, far in excess of that of conventional metal powders. Metal powders as heretofore made are solid and therefore expose only an exterior surface. The powder of the invention, on the other hand, is in the form of hollow spherical particles. The interior surfaces and exterior surfaces together provide an enormous amount of available surface for use. This is particularly important, for example, in the case of magnesium powder used for flares. Due to the enormous amount of available surface area the magnesium powder may be burned in a highly intense instantaneous flash.

It is at present believed that the particles are formed as the result of the manner in which the inert gases are employed. As noted above, a portion of the inert gas is withdrawn from the circulatory system and conducted to and over the body of molten metal in the furnace under substantial positive pressure, in order to force molten metal from the furnace to the atomizer. As a result of this step the molten metal becomes saturated with the gas at the prevailing temperature and pressure in the melting furnace, causing a certain amount of the gas to go into solution in the molten metal. When the molten metal is atomized, the resulting minute droplets are promptly frozen by the cool circulating inert gas into solid particles. The chilling effect is so rapid that the gas dissolved in the droplets is quickly forced out of solution thus causing them to expand into particles.

These and other features of the invention will be better understood by referring to the accompanying drawings, taken in conjunction with the following description, in which:

Fig. 1 is a diagrammatic flow sheet showing the system as a whole;

Fig. 2 is a side elevation of the purifier shown in Fig. 1;

Fig. 3 is a plan view of one of the trays in the purifier;

Fig. 4 is a cross-section on the line 4—4 of Fig. 3;

Fig. 5 is a cross-sectional view of one of the melting furnaces shown in Fig. 1;

Fig. 6 is an enlarged cross-sectional view of the atomizer shown in Fig. 5;

Fig. 7 is a longitudinal section of the chamber shown in Fig. 1 for atomizing molten metal and forming metal powder;

Fig. 8 is a transverse section on line 8—8 of Fig. 7;

Fig. 9 is an enlarged side view of one of the powder-gas separators shown in Fig. 1;

Fig. 10 is a similar view, partly in section, of the lower end thereof; and

Fig. 11 is a front elevation of the same.

Reference is made to Fig. 1 for an overall picture of the apparatus. Since purification of the gas in the system is of initial importance, the main features of its system or circuit will first be described.

This includes a gasometer 1, a conduit 2, a main conduit 3, a branch conduit 4, a dust filter 5, a conduit 6, a branch conduit 7, a filter 8, a branch conduit 9, a branch conduit 10, a filter 11, a branch conduit 12, a compressor 13 connected on its inlet or suction side with conduit 6, a conduit 14 connecting the outlet or pressure side of the compressor, a filter 15, a conduit 16, a branch conduit 17 connecting the inlet of a purifier 18 surrounded by a heating furnace or chamber 19 having an inlet 20 for the introduction of heating gases and an outlet 21 for the escape of spent gases, and a conduit 22 connecting the outlet of the purifier with the gasometer. The latter conduit may, of course, connect the system at any other suitable point. While some valves or closure members are indicated, it will be clear that any suitable number may be employed at other points in the circuit. In general, it will be seen that gas may pass from the main system by way of the gasometer and main conduit 3 to and through dust filter 5, one or both filters 8 and 11, to and through the compressor, through filter 15, purifier 18 and back into the system. This cyclic operation may be continued until all of the gas in the system reaches the desired purity. What has been described may be regarded as part of the main system, or indeed a circulating system within the main circulating system. When all of the gas is purified, it is unnecessary to continue with the purification

operation and it may be shut-off until again needed.

The main system or circuit for producing the metal powder will now be traced. It includes a branch conduit 30 connecting main conduit 3 with a bonnet 31 at one end of a molten metal atomizing and powder forming chamber 32 for providing cool inert gas with which to freeze the atomized metal to powder. Another branch conduit 33 also connects main conduit 3 with the lower portion of the chamber in order to supply additional gas for keeping the newly formed metal powder in suspension, as will be described in more detail below. A branch conduit 34 similar to branch conduit 30, connects main conduit 3 with a bonnet 35 at the other end of the chamber. Conduit 36 connects the same or discharge end of the chamber with a blower 37, a conduit 38, a powder-gas separator 40, a conduit 41, another powder-gas separator 42, a conduit 43, and a filter 44 which in turn connects main conduit 3. A branch conduit 45 connects the main conduit with a filter 46 and a conduit 47. Similarly a branch conduit 50 connects the main conduit with a filter 51 and conduit 52. In practice it is customary to alternate use of filters 46 and 51, so that while one is being cleaned or otherwise serviced, the other is used.

The next system or circuit to be traced is the one having to do with the use of gas to force a fine stream of molten metal to the atomizer and the use of gas to atomize the stream of molten metal. It includes conduit 16, from the outlet or pressure side of compressor 13 and a branch conduit 60 terminating in branch conduits 61 and 62. Branch 61 connects with a conduit 63 one end of which connects with the interior of a melting furnace 64 and the other end of which connects with the interior of chamber 32. A bonnet 65 connects the furnace to bonnet 31 of the atomizing and powder forming chamber.

A similar arrangement is provided for the other end of the chamber and includes a branch conduit 70 connecting conduit 16 and terminating in branch conduits 71 and 72. Branch 71 connects with a conduit 73 one end of which connects with the interior of a melting furnace 74 and the other end of which connects with the interior of chamber 32. A bonnet 75 connects the furnace to bonnet 35 of the atomizing and powder forming chamber.

Figs. 2, 3 and 4 show the purifier in detail. It is cylindrical in form with a closed bottom 80 (see Fig. 2) resting on supports 81 on the bottom of the interior of the heating chamber. Superposed trays or baskets 82 rest on an inner flange support 83 integrally secured to the side wall of the purifier, the flange being located directly above inlet 17. An imperforate cover 85 is securable to the top of the purifier by means of a plurality of bolts 86 through an outer flange 87. All joints of the purifier are gas tight so that heating chamber gases cannot enter the purifier and so that gases inside the purifier cannot escape into the heating chamber.

The trays (see Figs. 3 and 4) are generally cylindrical in shape, being formed of a band of sheet metal 90 with a turned in bottom flange 91 and a similar turned in top flange 92. A circular piece of coarse mesh circular screen 94 rests on and is secured to the bottom flange. A circular piece of fine mesh screen 95 in turn rests on the coarse mesh screen, the latter serving as a firm support for the former. A relatively thin layer of finely divided metal particles 96, such as magnesium powder, if magnesium powder is to be produced, rests on the screen. A handle or bar 97 is secured to the underside of upper flange 92.

In preparing the purifier for use, the top of heating chamber 19 is removed, cover 85 of purifier 18 is also removed and loaded trays or baskets 82 are placed therein, one superposed on the other. In the case of the bottom tray, its bottom flange 91 rests on inner flange support 83. Lower flange 91 of the second tray will rest on upper flange 92 of the first tray, etc. The purifier is provided with a sufficient number of the loaded trays. Cover 85 is then returned and bolted securely to outer flange 87 with bolts 86; after which the top of the purifier is returned.

Melting furnace 64 is shown in detail in Fig. 5. It rests on a dolly 90 so that it may be wheeled into and away from operating position with respect to chamber 32. The furnace is formed of a rectangular heating chamber 91 surrounded by a metal casing 92 lined with refractory brick 93. The chamber is surmounted by a removable top 95. A plurality of spaced openings 97 are provided through the side walls near the bottom of the chamber into each of which is fitted an oil or gas burner 98. Three such burners are at present employed.

A stack 99 is mounted on the top for the escape of spent heating gases. A melting pot 100 rests on supports 101 at the bottom of the heating chamber. The top of the melting pot is provided with a removable cover 102 securable to an outer flange 103 by means of a plurality of bolts 104 to provide a non-leaking joint. A metal charging conduit 106 extends through the top of the heating chamber and communicates with the interior of the melting pot. It is secured to cover 102 by means of a plurality of bolts 107. A removable cover 110 fits over the top of the charging conduit and can be screwed thereon to make a leak-proof joint. Branch conduit 63 for inert gas connects with the charging conduit above furnace top 95. A pyrometer 112 extends through the top and cover 102 well into the interior of the melting pot. Branch conduit 62 for inert gas extends into heating chamber 91 and is spirally wound around the melting pot. The discharging end of the conduit extends into the interior passageway 114 of brick-lined furnace bonnet 65. A conduit 115 for the passage of molten metal extends from near the bottom of the melting pot upwardly through its cover 102 and into passageway 114 of furnace bonnet 65 where it connects with an atomizer 116 extending through end-cover 117 into chamber bonnet 31. The two bonnets are connectible with bolts 119. The furnace bonnet is provided with an opening 120 in close proximity to the atomizer and is fitted with a burner 122 adapted to supply heating gases at and around the atomizer.

Atomizer 116 is shown in more detail in Fig. 6. It is in a form of a main body portion 124 hollowed out at one end to receive the discharge end of conduit 115 from the melting pot and hollowed out at the other end to receive an atomizing nozzle 125 and to form a gas distributing chamber 126 around the nozzle. The tip end of the nozzle, with discharge outlet 128, extends through cap 129 screwed onto the other end of the main body portion. Conduit 62 for inert gas connects with a nipple 130 communicating with the gas distributing chamber. Cap 129

is provided with a plurality of inclined circumferentially spaced discharge ports 132 adapted to blast small streams of preheated inert gas against a stream of molten metal discharged through nozzle outlet 128 at a point or area 134 a suitable distance forward of the nozzle tip.

Figs. 7 and 8 show chamber 32 for atomizing molten metal and forming powder therefrom in more detail. It is in the form of a cylindrical tank divided essentially into an upper compartment 140, which is in the form of a trough, a lower compartment 141, which is essentially in the form of a duct; and two side compartments 142 and 143, which are separated from each other as well as from the other two compartments. All of the compartments extend longitudinally of the chamber. The upper and lower compartments are tapered in reverse order. Thus, upper compartment 140 has its smallest cross-section at the right end of the tank, and its largest cross-section at the left end. The lower compartment or gas distributing duct 141, on the other hand, has its largest cros-section at the right end, where the gas is shown to enter through conduit 33, and its smallest cross-section at the left end.

The compartments are obtained by the use of opposed pairs of overlapping plates 151 to 158, top plates 161 to 168 and a pair of spaced side plates 171 and 172. They are suitably welded to the cylindrical wall and to each other to provide an integral structure. Plates 151 to 158 are pitched or slanted at an angle, toward the bottom of the trough, so that powder settling thereon will tend to slide down into the bottom of the trough. Referring for a moment to Fig. 7, it will be seen that an overlying lip 174 is welded to the head of the cylindrical tank at the right end to provide a slot 175 between inlet conduit 33 and upper compartment 140. Similar underlying lips 176 to 182 provide slots 185 to 191. Upper plate 161 of the duct is spaced above the bottom of the tank to provide a similar slot 192 at the left end of the tank, in direct communication with outlet conduit 36. The overlying and underlying lips are sufficiently long to cause gas passing therethrough to sweep along the bottom of the upper compartment. In other words, gas going through slot 175 sweeps along the top of plate 168, gas passing through slot 185 sweeps across the top of plate 167, gas passing through slot 186 sweeps across the top of plate 166, etc.

A gas blow-off 195 is provided along the top of the tank. In case of an explosion, the blow-off is opened to release explosive forces, thus saving the chamber from damage. An inlet 198 connects each side compartment with inlet conduit 33. An outlet 199 connects each side compartment with outlet 36. While the side compartments are normally sealed from each other and the other compartments, it is possible for leaks to develop at the welded joints. It is therefore desirable to have an arangement which will permit the side compartments to be cleared of air and filled with inert gas, and this can be done with the inlets and outlets referred to.

Figs. 9, 10 and 11 show powder-gas separator 49 in some detail. Powder-gas separator 42 is advantageously of the same general construction.

Referring first to Fig. 9, the apparatus shown is in the form of a combination powder-gas separator and collector, being specifically a conventional cyclone 200, with an inlet 201 for powder-gas and an outlet 202 for gas, and a hopper 203 integrally secured to and depending from the cyclone. The lower end of the hopper terminates in a laterally extending discharge conduit 205 at or near the outlet end of which is an upright relief chamber 206. A closure member 207 is pivotally supported at the outlet.

Referring next to Fig. 10, it will be seen that relief chamber 206 provides a substantial amount of free space 210 at its upper end which extends a suitable distance above the place where the chamber joints the conduit. The top of the relief chamber is fitted with a screw cap 211 provided with a plurality of circumferentially spaced handles 212.

In the specific construction shown the discharge outlet is at a convenient angle to cooperate with closure member 207, which is in the form of an enclosed chute pivotally or hingedly connected at 213 to a fixed support 214. The chute has a bottom 215, back 216, side walls 217 and 218, a top 219 and a discharge opening 220. A piece of resilient gasket material 222, such as rubber, is integrally secured to the bottom of the chute so that it may be brought into sealing contact with the discharge outlet.

The discharge outlet is in the form of a pipe welded at its side to the main part of the conduit, the upper end of the pipe being an extension which forms the relief chamber. The chute is integrally secured at its top 219 to an outer collar 224 fitting loosely around the lower end of the discharge conduit. The upper end of the collar is integrally secured to the lower end of a flexible sleeve 225 fitting loosely around the discharge conduit. This sleeve may be of rubber and is adapted to act like a bellows. The upper end of the flexible sleeve is in turn integrally secured to the discharge conduit. When the chute is moved upwardly and downwardly, the flexible sleeve yields sufficiently to permit the discharge outlet to be closed and opened.

A handle 228 is secured to the top of the chute at its discharge end so that the operator may readily lower the chute to open the discharge outlet and raise the chute to close the outlet. This is a manual operation which may remain wholly in control of the operator.

If the operator, however, should instinctively let go of the handle in case of fire, or for any other reason, or should leave the scene, self-closing means associated with the chute at once take care of such a contingency. The particular self-closing means disclosed include a pair of spring tensioning devices 230 and 231 secured at their lower ends to the chute and at their upper end to a fixed support, in the instant construction at the upper portion of the relief chamber. A turnbuckle is attached to each spring so that the springs may be placed under the proper amount of tension. When downward pressure is applied to the chute, such as by bearing down on handle 228, the springs yield sufficiently to permit the chute to drop away angularly from the discharge outlet. To make certain that the chute is not lowered unduly, for example, by the operator when excited, a chain 234 of predetermined length is fastened at its lower end to the chute and at its upper end to a fixed support. The chain fixes and limits the amount of drop for the chute. As a further precaution, a holding device 236 with a turnbuckle 238 is hooked at its lower end to the chute and at its upper end to a fixed support (not shown). When the chute is in its closed position, the turnbuckle is tightened. Pressure on handle 228 will, therefore, not open the chute. This feature is particularly desirable

if children or non-trained operators have access to the apparatus.

When, therefore, it is desired to discharge metal powder from the apparatus, the operator must deliberately loosen turnbuckle 238 in order to release holding device 236. He then applies downward pressure on handle 228, which causes chute 207 to move or open downwardly. Metal powder then moves by gravity from hopper 203 through conduit 205 and its discharge outlet into the chute, and downwardly through the chute out of its discharge opening 220 into a container 240.

If all goes well the handle is kept down until the desired amount of powder runs into the container. The handle is then lifted by the operator to close the discharge outlet and the holding device is again replaced. With the self-closing means in use, however, the operator need merely release the handle and spring-tensioning devices 230 and 231 will lift the chute into its closed position. As already indicated this feature is particularly desirable in case of fire and if the operator should let go of the handle or leave the scene in fright. In case of fire this is precisely what he should do for safety.

In practicing the method of the present invention, the apparatus may be operated as follows:

Since the circulatory system is initially filled with air, it is important that it be replaced with a suitable inert gas, such as helium. Helium for this purpose is obtained commercially in cylinders. With an adequate supply of cylinders on hand, the helium is fed into the system at one or more high points and air is removed therefrom at one or more low points; for example, referring to Fig. 1, the helium may be fed into gasometer 1 and air may be withdrawn from conduit 36 in advance of blower 37. Whatever practice is followed a sufficient amount of helium is fed into the system to flush out most of the air. Enough helium is thus used to place the system under substantial positive pressure greater than atmospheric to keep outside air from seeping into the system. A manometer pressure of 2'' water has worked well.

Since helium and air are readily miscible, no matter what precautions are taken an appreciable amount of air will be in the system after the flushing operation; and since this residue of air and the helium contain objectionable amounts of oxygen and nitrogen, purifier 18 is placed in operation. As shown in Fig. 1 circulation of the helium through the system may be effected by the use of compressor 13, as well as by blower 37, or both. In any event helium from the system is passed continuously and cyclically through the purifier. Heating gases, such as provided by oil or gas burners, are fed through inlet 20 into heating chamber 19, while spent heating gases escape from the heating chamber through outlet 21 to the open atmosphere. Impure helium from the system is forced by compressor 13 through conduit 14, filter 15, conduit 16 and branch conduit 17 into the purifier 18. The purified helium rises upwardly through conduit 22 and is returned to the main system.

As more particularly shown in Figs. 2, 3 and 4, the impure helium passes upwardly through layers 95 of finely divided metal particles in superposed perforated trays 82. The heating gases applied externally to the purifier are adapted to heat the metal particles to a temperature sufficiently high for the oxygen and nitrogen impurities to react therewith to form metal oxide and metal nitride. Assuming that magnesium powder is to be produced, the layers are formed preferably of magnesium powder, about ¼'' to ½'' deep; the depth being such that the helium may pass readily therethrough. The oxygen and nitrogen impurities react with the magnesium to form magnesium oxide and magnesium nitride which are retained in the layers on the trays. In the particular arrangement shown, the helium thus purified (see Fig. 1) passes through conduit 22 into gasometer 1 where it mingles with helium still to be purified.

Since the circulatory system is open, helium passes continuously through gasometer 1, conduits 2, 3 and 4, filter 5, conduit 6, branch conduit 7, filter 8, branch conduit 9, branch conduit 10, filter 11, branch conduit 12, compressor 13, conduit 14, filter 15, conduit 16, branch conduit 17, purifier 18, conduit 22, and again gasometer 1. As this cyclic operation continues, helium is also forced by blower 37 through conduit 38, powder-gas separator 40, conduit 41, powder-gas separator 42, conduit 43, filter 44, main conduit 3, branch conduit 45, filter 46, conduit 47, as well as branch conduit 50, filter 51, conduit 52, main conduit 3, branch conduits 30 and 33 into and through chamber 32, inlets 198, side compartments 142 and 143, outlets 199, conduit 36, and back to blower 37.

In a similar manner compressor 13 also pulls some of the helium from main conduit 3 through branch conduit 4, dust filter 5, conduit 6, branch conduit 7, filter 8, branch conduit 9, as well as branch conduit 10, filter 11, branch conduit 12, conduit 6, the compressor itself, conduit 14, filter 15, conduit 16 past branch conduit 17 to and through branch conduit 60, branch conduits 61 and 62 to furnace 64. As more clearly shown in Figs. 5 and 6, helium passing through conduit 61 goes into melting pot 100. This helium may be passed through conduit 63 into chamber 32. The helium passing through branch conduit 62 moves through the coils surrounding the melting pot and is passed into and through atomizer 116, after which it mingles with helium passing through chamber 32.

If furnace 74 is also placed in operating position with respect to chamber 32, its melting pot may be flushed of air and filled with helium undergoing purification in a manner similar to furnace 64. Helium is forced by the compressor through conduits 16 and 70, and branch conduits 71 and 72. Helium passing through conduit 71 into the melting pot may be passed through conduit 73 into chamber 32. Helium passing through branch conduit 72 is passed through the coils around the melting pot and finally through the atomizer into chamber 32.

It will be clear from what has been said that if the impure helium in the system is circulated for a sufficiently long time, it will continue to pass through the purifier until substantially all of the objectionable oxygen and nitrogen are removed. Periodic tests are made, with an Orset tester, to determine the oxygen content of the helium. When it is sufficiently pure the valves in inlet 17 and outlet 22 are closed to cut-out the purifier circuit. It may be cut-in from time to time as needed. If all goes well, the purified helium may be retained in the system and used over a prolonged period of time.

With the circulatory system full of purified helium, it is ready for the actual metal powder producing operation. Referring for the moment to Fig. 5, which shows melting furnace 64 and its

auxiliary equipment in more detail, cover 110 is removed from charging conduit 106. Ingots of metal to be converted into metal powder are dropped into melting pot 100. After a suitable amount is placed therein the cover is returned. The apparatus is at present being used to produce magnesium powder and also magnesium-aluminum alloy powder. The inside dimensions of the pot are 24" in diameter and 42" high. A charge of about 180 pounds of magnesium is thus introduced. Oil burners 98 are operated to heat the pot externally until its magnesium content reaches a suitable molten temperature, from 1300° to 1350° F. The burners are kept in operation to maintain this temperature.

Pressure-gas conduit 61 is opened to admit helium into the charge conduit and top portion of the pot above the level of the molten magnesium. Since the pressure-gas conduit communicates with the compressor the molten magnesium is placed under sufficient gas pressure to force a stream of magnesium upwardly through molten-metal conduit 115 to and through atomizer 116 into atomizing and powder forming chamber 32. A pressure of 12–15 pounds per square inch is applied initially to the top of the body of molten magnesium to start its passage through the conduit and the atomizer. After atomization of the metal is underway the pressure is reduced to about 5 pounds per square inch. Helium under adequate pressure by the compressor is passed through atomizing-gas conduit 62 where it is heated in the coils around the melting pot. The preheated helium is passed through nipple 130 (see Fig. 6) into gas distributing chamber 126 of the atomizer, from which it issues through discharge ports 132 of cap 128 in a plurality of streams or blasts and atomizes the fine stream of molten magnesium discharged through nozzle outlet 129 at point or area 134 a suitable distance forward of the nozzle tip. The pressure of the helium at the nozzle varies somewhat according to its construction. A pressure in the neighborhood of 60 pounds per square inch works satisfactorily with the nozzles now employed.

Returning for a moment to Fig. 1, it will be recalled that blower 37 causes the helium to circulate continuously through the system. Since chamber 32 is on the suction side of the blower, the helium is in effect sucked through and from the chamber only to be forced or pushed from the pressure side of the blower around the system. Relatively cool helium enters the chamber by way of branch inlet conduits 36 and 33 at the same end, adjacent melting furnace 64. The atomized magnesium is promptly enveloped and frozen into powder particles in the cool helium sweeping through upper compartment 140 (see Figs. 7 and 8).

While the newly formed particles of magnesium powder tend to remain in suspension in the helium as the helium passes through the chamber to the blower, some of it also tends to settle by gravity onto plates 151 to 158 and toward the bottom or base of the trough of the upper compartment. Additional amounts of relatively cool helium are, therefore, introduced into the upper compartment by way of inlet conduit 33. According to a present practice, the amount of helium entering the upper compartment by way of inlet 33 and lower compartment or duct 141 substantially exceeds that entering by way of inlet conduit 36. Some of the helium from inlet 33 issues as a relatively small stream through slot 175, the stream being directed along the top of plate 168 in the bottom of the trough. Any magnesium powder settling or tending to settle on that plate is, therefore, swept up and placed in suspension in the main and large current of gas or helium passing through the upper compartment.

In a similar manner some helium in lower compartment or duct 141 passes as a stream through slot 185 along the top of plate 167 at the bottom of the trough of the upper compartment. Similar streams of helium pass through slots 186 to 191, so that plates 166, 165, 164, 163, 162 and 161 at the bottom of the trough of the upper compartment are continuously swept with streams of helium under sufficient velocity to keep the powder from settling and to keep it in suspension. An additional stream of helium passes through duct 192 into the discharge end of the upper compartment. Some of the helium thus introduced through the slots also spreads to the side and sweeps across plates 151 to 158 to keep them clean of powder. The mixture of gas and powder is under a great deal of turbulence which inhibits settling of the powder. The powder laden gas is in effect sucked from the upper compartment through outlet 36 and blown through conduit 38.

If melting furnace 64 must be shut down for some reason, such as for repairs, melting furnace 74 (see Fig. 1) is put in operation at the other end of the chamber. In this case it is advisable to close inlet conduit 33 and to open inlet conduit 34. As before, a fine stream of molten magnesium issuing from the nozzle of the atomizer is blasted with helium and the atomized magnesium is promptly enveloped in the relatively cool helium gas entering the upper compartment by way of inlet conduit 34 as well as by helium entering the chamber through inlet conduit 33.

The force behind the fine stream of molten metal issuing from the nozzle of the atomizer at either end of the chamber and the force of the helium used to atomize the stream of molten magnesium is sufficient to throw the atomized magnesium substantially across the length of the chamber. When melting furnace 64 is in operation the direction of the stream of molten metal is in general concurrent with the streams of helium passed into and through the chamber. On the other hand, when melting furnace 74 is in operation the direction of the stream of molten metal issuing from the nozzle is in general countercurrent to the helium entering the upper compartment by way of inlet conduit 33 but concurrent with the helium entering the chamber through inlet conduit 34. As a result of these contrary movements, at least initially, the helium is kept in a turbulent state. In any event the suction force of the blower is sufficient to draw the resulting powder-gas mixture from the chamber.

Still referring to Fig. 1, the magnesium powder laden helium is forced by blower 37 through conduit 38 to powder-gas separator 40. As shown in Figs. 9, 10 and 11 and as described in some detail above, the larger powder particles are separated selectively from the helium and smaller particles in cyclone 200 and fall into hopper 203. After a sufficient supply of powder has collected in the hopper, some of it is withdrawn from time to time; care being taken not to break the powder seal in discharge conduit 205 so that air can enter the system.

Again returning to Fig. 1, the helium containing the smaller particles of magnesium powder is forced from powder-gas separator 40 through

conduit 41 to powder-gas separator 42. Since the separator operates like the other, the same procedure is followed in withdrawing powder. As already indicated one or more additional powder-gas separators may be employed.

From the chamber to and through the powder-gas separators the helium undergoes a substantial drop in temperature. As it leaves the last powder-gas separator in the series it still contains some fines which should be removed before it reaches the compressor. To this end the helium leaving powder-gas separator 42 is passed through conduit 43 into dust-collector 44. In the present practice this is in the form of a plurality of filter bags, the specific device employed being a Dracco dust collector. While a substantial amount of the dust is thus removed from the helium, some remains.

Helium leaving dust-collector 44 passes into main conduit 3 and is then diverted through branch conduit 45, filter 46 and conduit 47 back to main conduit 3. This is a wet filter, of the oil type, which needs to be cleaned from time to time. When cut-out of the circuit for that purpose, helium is diverted from main conduit 3 through branch conduit 50, a similar filter 51 and conduit 52 back to main conduit 3.

A substantial amount of the helium thus treated passes continuously through main conduit 3 and branch conduits 30, 33 and 34 back to chamber 32. Some of the thus treated helium is, however, diverted from main conduit 3 to the compressor. Additional steps are taken to remove further amounts of dust from this helium before it reaches the compressor. To this end the helium is diverted through conduit 4 into and through filter 5, which may be an oil filter similar to filters 46 and 51. Although the helium thus treated is substantially dust free, it usually contains some suspended oil and moisture, both of which contribute to the fire hazard. Helium leaving the filter enters conduit 6 and is diverted through branch conduit 7, filter 8 and conduit 9 back to conduit 6 which connects the inlet or suction side of compressor 13. When filter 8 is cut-out for cleaning, helium leaving filter 5 is diverted from conduit 6 through branch conduit 10, filter 11, and conduit 12 back to conduit 6. Filters 8 and 11 are advantageously of the pot type containing a filter layer of steel wool or other suitable material, through which the helium is passed to abstract the suspended oil and moisture.

Since the helium may pick up some oil and moisture in the compressor, it is again filtered. Helium leaving the outlet or pressure side of the compressor is passed through conduit 14 and filter 15. This filter may contain a filter layer of felt, for example, such as a Cuno filter, which abstracts the oil and moisture as the helium passes therethrough. The helium thus treated for the removal of powder, fines, oil and moisture is passed through the remainder of the system, including the purifier when cut-in, the melting pots, and the atomizers, as already described.

Some losses of helium from the system are unavoidable, not only from minor leaks but also when charging the melting furnaces, etc. Replacement helium must, therefore, be added to the system. However, by keeing the system under positive pressure, ingress of air is for the most part avoided. Such oxygen and nitrogen as enter by replacement helium is so small compared with the total volume of helium in the system that their effect is of little consequence. They react with the atomized magnesium or highly heated powder in the chamber and are quickly eliminated. That is, after the system is in metal powder producing operation, it acts to purify itself so far as the extremely small amounts of oxygen and nitrogen are concerned.

It will thus be seen that a confined system may be provided in which an inert gas of high purity under positive pressure is continuously circulated while metal to be converted into powder is melted and atomized therein. Although the gas initially placed in the system is not satisfactory for the purpose, it may be so treated as to remove harmful impurities. After the gas is purified, it may be used over a long period of time to produce metal powder. While various inert or non-reactive gases may be employed, helium is especially suitable because of its availability. Metal powder may be produced from various metals and their alloys, such as magnesium, aluminum, zinc, cadmium, lead, etc. The principal limitation is the ability of the materials in the system economically to withstand the necessary wear and tear.

It will be clear to those skilled in this art that the practice of the invention lends itself readily to various modifications. The specific practice described is only by way of illustration. It is obvious, for example, that other compressors, purifiers, furnaces, atomizers, separators, filters, etc., could be used and that the units making up the whole can be arranged in various ways. What is disclosed is a highly effective arrangement for producing metal powders in large quantities in an efficient manner.

I claim:

1. In the method of producing metal powder by atomizing molten metal with a blast of inert gas, freezing the atomized metal into finely divided solid particles, and recovering the resulting metal powder, the improvement which comprises conducting the molten metal atomizing, metal powder-forming and powder-separating steps in successive zones therefor of a main confined circulatory system filled with gas inert to the metal, circulating the gas continuously therethrough under a pressure higher than atmospheric to prevent ingress of outside air, withdrawing continuously a relatively small portion of the inert gas from the main circulatory system beyond the powder-separating zone and in advance of the atomizing and powder-forming zones, compressing the gas so withdrawn substantially to increase its pressure above that at any point in the main circulatory system, and atomizing the molten metal in the main circulatory system with at least a part of the compressed gas, the compressed gas being returned to and mixed with the gas in the main circulatory system.

2. Method according to claim 1, in which the circulating inert gas with the newly formed metal powder suspended therein is passed from the powder forming zone through a plurality of powder-gas separating zones, metal powder is separated from the inert gas in each latter zone, and the metal powder so separated is withdrawn from the system at each latter zone.

3. Method according to claim 1, in which the inert gas so withdrawn is specially heated, a continuous fine stream of the molten metal is blasted circumferentially with a plurality of streams of the heated inert gas in a metal powder forming zone filled with the main body of circulating inert gas, the main body of gas being suf-

ficently low in temperature promptly to freeze the atomized metal into finely divided particles, the main body of circulating inert gas with the newly formed metal powder suspended therein is passed from the powder forming zone to a powder-gas separating zone, metal powder is separated from the main body of inert gas, and the metal powder so separated is withdrawn from the system.

4. Method according to claim 1, in which the main body of circulating inert gas with metal powder suspended therein is passed from the powder forming zone to a powder-gas separating zone, metal powder is separated from the main body of inert gas by dry precipitation, and the metal powder so separated is withdrawn from the system.

5. Method according to claim 1, in which the main body of circulating inert gas with metal powder suspended therein is passed from the powder forming zone to a powder-gas separating zone, metal powder is separated from the main body of inert gas by wet precipitation, and the metal powder so separated is withdrawn from the system.

6. Method according to claim 1, in which the main body of circulating inert gas with metal powder suspended therein is passed from the powder forming zone successively through a series of at least two powder gas separating zones, coarser metal powder being separated from the main body of inert gas in an earlier zone by dry precipitations, and finer metal powder being separated from the main body of inert gas in a later zone by wet precipitation.

7. Method according to claim 1, in which some of the inert gas temporarily withdrawn from the system and compressed is heated before being used to atomize the molten metal.

8. Method according to claim 1, in which some of the inert gas withdrawn from the system and compressed is conducted while under substantial positive pressure to a confined space above a body of the molten metal to be atomized and used to force a stream of the molten metal to the atomizing zone.

9. Method according to claim 1, in which the gas in the system is purfied with respect to oxygen and nitrogen by passing it through a body of finely divided particles of heated metal adapted to react with the oxygen and nitrogen to form metal oxide and nitride.

10. Method according to claim 1, in which gas in the system contaminated with oxygen and nitrogen is withdrawn from the system, passed through a body of heated metal powder adapted to react with the oxygen and nitrogen, and the purified inert gas is returned to the system.

11. Method according to claim 1, in which gas in the system contaminated with oxygen and nitrogen is passed through a plurality of thin beds of finely divided particles of heated metal adapted to react with the oxygen and nitrogen.

12. Method according to claim 1, in which gas initially in the system and contaminated with an objectionable amount of oxygen and nitrogen, is first passed through a body of finely divided particles of heated metal adapted to react with the oxygen and nitrogen until the gas is purified with respect to the oxygen and nitrogen, and the atomizing, metal powder forming and powder separating steps are then conducted in the presence of the inert gas so purified.

13. Method according to claim 1, in which a fine stream of the molten metal is blasted with inert gas so withdrawn in a metal powder forming zone filled with the main body of circulating inert gas, the main body of circulating gas being sufficiently low in temperature promptly to freeze the atomized metal into finely divided solid particles, and a stream of the inert gas withdrawn from the main body of gas is introduced at the bottom of the powder forming zone to help keep the newly formed particles of metal powder in suspension during their passage to the powder separating zone.

14. Method according to claim 1, in which the inert gas so withdrawn is specially heated, a fine stream of the molten metal is blasted with the heated inert gas in a metal powder forming zone filled with the main body of circulating inert gas, the circulating gas being sufficiently low in temperature promptly to freeze the atomized metal into finely divided particles, a plurality of streams of inert gas from the main circulatory system is introduced at spaced intervals along the bottom of the powder forming zone to help keep the newly formed particles of metal powder in suspension during their passage from the powder forming zone to the powder separating zone.

15. Method according to claim 1, in which a fine stream of the molten metal is blasted with the inert gas so withdrawn in one end of an elongated metal powder forming zone filled with the main body of circulating inert gas, the main body of circulating gas being sufficiently low in temperature promptly to freeze the atomized metal into finely divided particles, and a plurality of streams of inert gas from the main circulatory system is introduced along the bottom of the powder forming zone to help keep the newly formed particles of metal powder in suspension during their passage from the powder forming zone to the powder separating zone.

16. Method according to claim 1, in which the inert gas so withdrawn is specially heated, a fine stream of the molten metal is blasted circumferentially with a plurality of fine streams of the heated inert gas so withdrawn in one end of an elongated metal powder forming zone filled with the main body of circulating inert gas entering therein from the same end, the main body of circulating gas being sufficiently low in temperature promptly to freeze the atomized metal into finely divided particles, introducing the main body of circulating inert gas in the powder forming zone at the same end so that the powder and gas may move concurrently through the powder forming zone, and a plurality of streams of inert gas from the main circulatory system is introduced along the bottom of the powder forming zone to help keep the newly formed particles of metal powder in suspension during their passage from the powder forming zone to the powder separating zone.

17. Method according to claim 1, in which the gas so withdrawn from the main circulatory system is filtered to remove solids therefrom before it is compressed.

18. Method according to claim 1, in which a portion of the gas so compressed is used to force the molten metal into the atomizing zone.

19. Method according to claim 1, in which a part of the withdrawn and compressed gas is purified with respect to oxygen and nitrogen by passing it through a body of finely divided particles of heated metal adapted to react with

the oxygen and nitrogen to form metal oxide and nitride.

20. Method according to claim 1, in which the gas withdrawn from the main circulatory system for compression is filtered to remove solids therefrom before being compressed, and at least a part of the compressed filtered gas is purified with respect to oxygen and nitrogen by passing it through a body of finely divided particles of heated metal adapted to react with the oxygen and nitrogen to form oxide and nitride.

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