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VACUUM VAPOR CONDENSATION PROCESS FOR PRODUCING FINE METAL POWDERS

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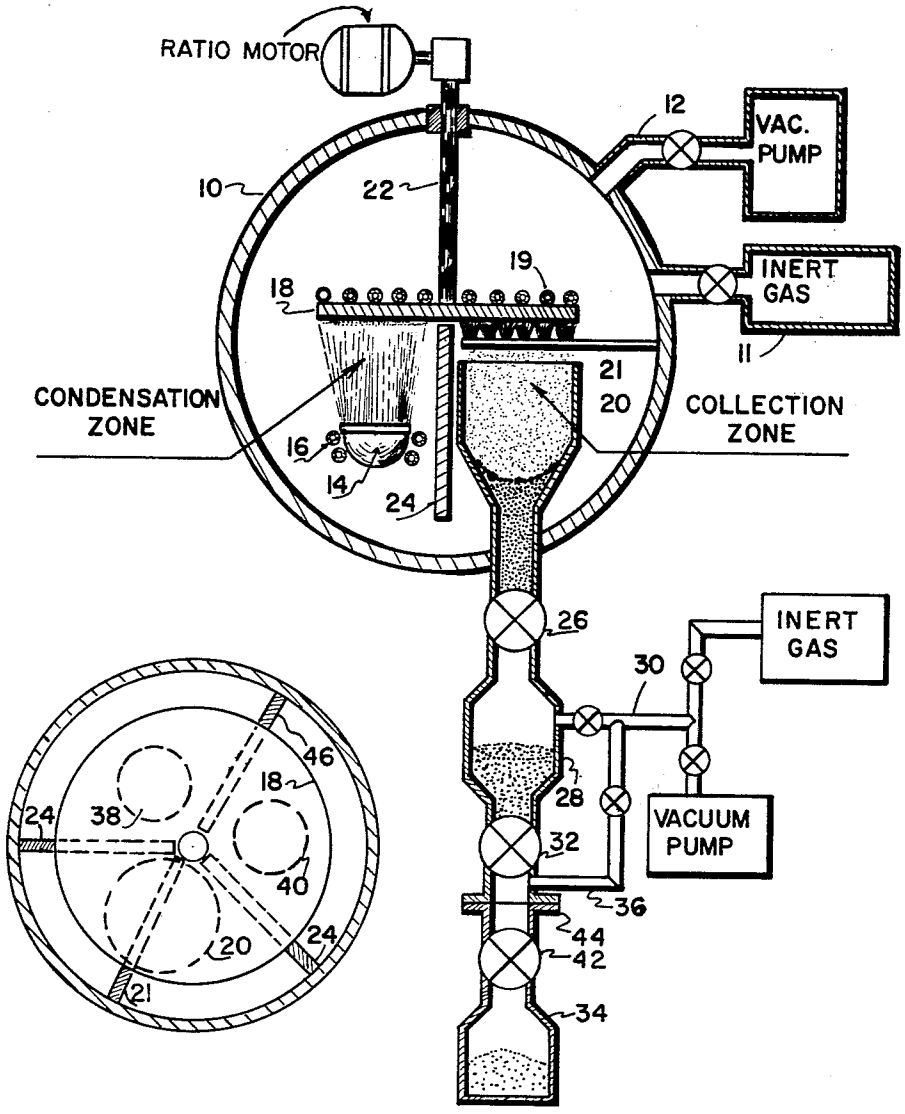


FIG. 1

FIG. 2

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VACUUM VAPOR CONDENSATION PROCESS FOR PRODUCING FINE METAL POWDERS

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This invention relates to the production of metals and more particularly to the production of extremely fine metal powders.

A principal object of the present invention is to provide an economical, simple, vacuum vapor condensation process for the production of extremely fine metal powders.

Another object of the invention is to provide a process of producing high purity metal powders in a relatively confined space.

A further object of the invention is to provide a relatively simple, economical apparatus for carrying out the above process.

A further object of the present invention is to provide a method of producing fine alloy powders and multimetallic powders.

A still further object of the invention is to provide a method of varying the particle size of fine powders.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the apparatus possessing the construction, combination of elements and arrangement of parts, and the process involving the several steps and the relation and order of one or more of such steps with respect to each of the others which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawing wherein:

FIG. 1 is a diagrammatic, schematic view of a preferred apparatus for practicing the invention; and

FIG. 2 is a diagrammatic, schematic top view showing a modification of the apparatus of FIG. 1 for practicing the invention.

The present process comprises thermally evaporating a metal selected from the group consisting of aluminum, manganese, silver, chromium, beryllium, copper, boron, silicon, iron, nickel, zinc, magnesium, titanium, zirconium, tantalum, tungsten, molybdenum, niobium, thorium and bismuth. The metal vapors produced are then condensed to a powder on a cooled surface positioned above the vapor source and the condensed powder is substantially immediately removed from a condensation zone to a collection zone. In the collection zone the powder is removed from the surface of the condenser and collected in a receiver. The process is preferably carried out in a substantially inert atmosphere, e.g. vacuum or an inert gas. The removal of the powder from the condensation zone is preferably accomplished by employing a moving cooled surface. The moving cooled surface is preferably a circular condenser plate positioned so that a portion of the plate is exposed to the condensing vapor and a portion is exposed to the collection zone. Thus the deposited metal powder can be removed from the condensing zone to the collection zone by rotating the plate. It is apparent that other types of moving cold surfaces, such as belts and the like, can be employed, the main requirements being that it be capable of cooling and rapidly removing the condensed powder from the condensation zone.

The postulated mechanism of the present process is based on the premise that, maintaining the cold plate at a sufficiently low temperature, the energy of the impinging metal vapors is sufficiently reduced to prevent substantial crystal growth. Thus, the cold surface is provided with an accommodation coefficient approaching 1. The term "accommodation coefficient" is defined as the ratio between the number of molecules which actually condense on the surface of the cold plate and the number of molecules which strike the surface of the cold plate. Thus, the accommodation coefficient is primarily a function of the temperature of the cold plate and the density of the incident molecules at the cold plate surface (i.e., the rate of evaporation). Accordingly, since the condensing vapors and the previously condensed particles lose their energy rapidly to the cold surface, the tendency to grow large crystals is enormously impeded. Thus, by drastically reducing the mobility of the condensing atoms and rapidly removing the condensed powder from the condensation zone, to prevent heating of the condensed powder by the vapor stream, with resultant crystals growth, very fine powder results.

Additionally it is further postulated that, if the layer of condensed powder is allowed to increase in thickness, the temperature of the surface contacted by subsequent impinging vapors will be the temperature of the surface of the outermost layer of condensed powder particles. Since this layer cannot transmit heat rapidly through any appreciable thickness of powder, the energy of the impinging vapors will not be sufficiently rapidly removed. The resulting surface atoms will then have sufficient residual thermal energy to provide adequate mobility for larger crystal growth and hence larger particle size. It can be seen then that reduction in speed of rotation of the cold plate (rate of removal from the condensation zone) and/or increased rate of evaporation will provide an increase in the thickness of layer of condensed powder and resultant larger crystal growth for a given cold plate temperature.

In this manner it is believed that the particle size of the condensed powder can be varied. By the term "particle size," as used in the specification and claims, it is meant the particle size as calculated from the surface area per unit weight of the metal powder assuming that the powder particles are spherical in shape.

In carrying out the process, a charge of the material to be evaporated, a metal for example, is placed in the crucible and heated to its vaporization temperature. The temperature required for evaporating the metal, of course, depends upon the vapor pressure of the particular metal and the operating pressures employed. The temperature, at which the evaporation is carried out, determines the rate of metal vapor efflux or rate of evaporation from the source containing the molten metal. The temperatures at which the vapor pressure of the metal is below approximately 0.1 millimeter of mercury will yield low evaporation rates while higher temperatures and correspondingly higher metal vapor pressure will yield higher evaporation rates. Thus, the rates of evaporation can be varied considerably.

The pressures employed for the metal evaporation and condensation of the metal vapor are preferably obtained by evacuating the system to an extremely low pressure on the order of 0.1 micron or lower and then adjusting to the desired operating pressure with an inert medium such as argon, helium and the like.

As the metal vapors emanating from the vapor source pass upwardly through the inert gas they come in contact with and are condensed upon the cooled condenser plate. The condenser plate is preferably cooled to a temperature on the order of 20° C. or below. The condensed powder is then substantially immediately carried from

the condensation zone by the condenser plate to the collection zone where a brush or knife is mounted to remove the condensed powder from the plate and drop it into receiving means.

When the high purity of the metal powders is to be retained, then collection thereof (which includes screening, if employed, storage, shipping and the like are conducted under non-oxidizing conditions. The various handling steps are carried out in an inert medium. For example, since the powders are produced in a substantially inert atmosphere, e.g. vacuum or inert gas, they can also be screened, packed and stored under vacuum conditions. The powders can also be handled and stored under an organic liquid or solid which will protect the powders from oxidation. Combinations of the various inert mediums described can be used too. For instance, the powder can be collected and screened under vacuum conditions and then stored and shipped in containers in a non-reactive organic liquid or solid.

In respect to the "particle size" of the metal powder, the present process has been found to be of further advantage in that by controlling the inert gas pressure at which evaporation and condensation of the metal takes place (for a given vapor source to condenser plate distance), the "particle size" of the metal powders produced can be varied. Increasing the inert gas pressure at which evaporation and condensation takes place provides for a powder having a larger surface area and smaller "particle size" while decreasing the pressure results in a powder having a smaller surface area per unit weight and a larger "particle size."

The mechanism by which the "particle size" of the powder is varied is believed to occur in the following manner. When the inert gas pressure is increased the metal atoms lose more energy through increased collisions with the inert gas molecules. The result is that the metal atoms upon reaching the condenser surface have less residual energy to provide adequate mobility for larger crystal growth. When lower inert gas pressures are employed less collisions occur with the result that the metal atoms have sufficient residual energy to provide larger crystal growth.

The present invention also has particular utility where it is desired to produce multi-metallic powders or alloy powders.

For example, a bi-metallic powder can be formed by positioning two metal vapor sources in the vapor zone of the vacuum tank so that the condenser plate will sequentially pass over the vapor sources. In this way the first metal powder deposit would be coated with the second metal deposit to produce a bi-metallic powder. The vapor sources are preferably suitably baffled to prevent intermixing of the metal vapors prior to condensation. Obviously, more than two vapor sources can be employed where multi-metallic powders are desired. By controlling the rate of evaporation and the effective evaporation area of the vapor sources, the composition of the condensed powder can be varied. Additionally, the particle size of the powder can be varied in the same manner as described hereinbefore.

Where it is desired to produce alloy powders, it is necessary that the vapors of the alloying elements be mixed prior to being condensed on the cooled surface. Intermixing of the constituent metals can be accomplished in diverse ways.

For example, the metal vapor sources can be arranged, as by tilting, so that the vapor streams emanating therefrom intercept and thereby intermix before reaching the cooled surface. Intermixing can also be effected by providing suitable baffles or deflecting surfaces which deflect the vapor streams along intercept paths.

One preferred type of equipment for producing the metal powders in accordance with the invention is shown in FIG. 1 of the drawing wherein **10** represents a cylindrical vacuum-tight tank or chamber which is evacuated through conduit **12** by means of a suitable pumping system. A source **11** of inert gas is provided for filling tank **10** with any desired amount of such inert gas. Within tank **10** there is a vapor source **14** here shown as a crucible means for holding a charge of the metal to be evaporated. The vapor source **14** is suitably heated by means **16** illustrated as an induction heating means. Obviously, other types of vapor sources and heating means than those shown can be employed.

A circular condenser plate **18** is positioned above the vapor source **14** and the powder collection hopper **20**. Condenser plate **18** is preferably adjustable to the desired height above the vapor source and is held in position and rotated by assembly **22** driven through a vacuum seal. Cooling means **19** through which a refrigerant such as water can be circulated are provided on condenser plate **18**. The cooling means **19** are preferably copper coils attached to the back of the condenser plate **18**. Condensed powders are preferably removed from the condenser plate by a metal brush means **21**. The metal brush **21** is preferably constructed of the same metal as the powder to be produced and in the case of multi-metallic powders the same metal as one of the constituent metals. Similarly, where alloy powders are produced, the brush is preferably constructed of an alloy similar to the alloy to be produced. A vertical baffle **24** is mounted so that one portion of the condenser plate **18** is exposed to the vapor source **14** and another portion shielded from the source and exposed to the powder collection hopper **20**.

Collection hopper **20** is preferably connected, by valve means **26**, to a powder collecting chamber **28**. A non-oxidizing medium is provided within chamber **28**. This is preferably accomplished by evacuating chamber **28** through conduit **30** to a low pressure by means of a suitable vacuum pumping system. A source of inert gas can be provided for filling chamber **28** with any desired amount of such inert gas.

Valve means **32** is provided between collecting chamber **28** and a storage or shipping receiver **34**. A non-oxidizing medium is created within receiver **34** utilizing conduit **36** in the same manner as conduit **30**. Suitable screening means, not shown, can be located between chamber **28** and receiver **34**. If such screening means are employed, then a plurality of receivers can be suitably coupled to collect the various sized metal powders.

In operation the powder which condenses on plate **18** is removed by brush means **21** as the plate is rotated. The powder thus removed settles to the lower portion of hopper **20**. Periodically, valve **26** is opened (valve **32** being closed) to permit an appreciable quantity of metal powder to fall into chamber **28** which is preferably maintained at a reduced pressure. Valves **32** and **42** are then opened (valve **26** being closed) to permit the powder to fall into receiver **34**. Receiver **34** and collection chamber **28** are preferably filled with an inert, organic liquid or inert gas or maintained under reduced pressure when the powders are pyrophoric and highly reactive. Screening of the metal powders can be done at any point. Valves **32** and **42** are then closed and chamber **28** is then prepared for the next batch of powder. The receiver **34** is then disconnected at flange **44** and removed. A new receiver is placed into position and prepared for use.

Referring now to FIG. 2 there is shown a top view of a modification of the apparatus of FIG. 1 for producing multi-metallic powders or alloy powders in accordance with the invention.

Within the condensation zone of tank **10** there is provided a vapor source **38** for holding a charge of a metal, for example, nickel, to be evaporated. Also positioned within the condensation zone of tank **10** is a second vapor source **40** for holding a charge of a different metal, for example, aluminum, to be evaporated. In the case of bi-metallic powders, a baffle **46** is positioned between

vapor sources 38 and 40 to prevent intermixing of the metal vapors prior to condensation.

Where it is desired to vary the proportions of the constituent metals, the proportion of the effective evaporation area of the vapor sources can be varied. For example, the vapor source 38 can be constructed to have a larger evaporation area than vapor source 40 and thus provide for a greater proportion of nickel than aluminum in the bi-metallic deposit. Also the proportions of the constituent metals can be varied by changing the rate of evaporation of the metals.

As the condenser plate 18 is rotated it would first pass over vapor source 38, baffle 46, and subsequently over vapor source 40. In this way the metal deposit of nickel would be coated with the aluminum metal.

The bi-metallic deposit would then be removed by brush 21 upon continued rotation of condenser plate 18 past the baffle 24 and through the powder collection zone. The powder removed can be collected in the same manner as described and shown with respect to FIG. 1.

It is to be understood that additional vapor sources and baffles can be provided where it is desired to produce powders composed of more than two constituent metals.

Where an alloy, for example, Al_3Ni_2 is to be produced, baffle 46 can be removed and the vapor sources 38 and 40 tilted to permit intermixing of the nickel and aluminum vapors prior to condensation on the cold plate 18. In other respects the procedure is the same as described above.

The invention will now be described by way of the following non-limiting examples.

Example 1

Aluminum was placed in the crucible 14 and the tank 10 was closed and evacuated to a pressure on the order of 0.1 micron Hg abs. to remove most of the residual gases. During the evacuation the induction heating coil 16 was energized and the aluminum brought up to melting temperature. During this period, the pressure was adjusted by bleeding in argon from inert gas source 11. When the desired pressure of about 50 to 60 microns had been obtained, the aluminum melt temperature was raised to about 1350° C. so as to cause evaporation of the aluminum.

The aluminum vapors passed upwardly through the atmosphere of argon and were collected on rotating cooled plate 18 positioned 2 inches above the aluminum vapor source. The plate was approximately 18 inches in diameter and water cooled to a temperature of about 10° C. The collector plate was rotated at about 100 r.p.m. A steel brush 21 removed the condensed powder from the plate and dropped it into the collection hopper 20. The aluminum powder produced was grey in color and was non-pyrophoric. The powder had a surface area of 7.6 square meters/gram and calculated particle size of 0.32 micron (micron=3.937×10^{-5} inches).

Example 2

This example was similar to Example 1 in all respects except that the evaporation and condensation of the aluminum was performed at a pressure of about 100–130 microns Hg abs. of argon. The aluminum powder produced was grey in color and was non-pyrophoric. The powder had a surface area of 8.6 square meters/gram and a calculated particle size of 0.28 micron.

Example 3

This example was similar to Examples 1 and 2 in all respects except that the evaporation and condensation of the aluminum was carried out at a pressure of about 180–200 microns Hg abs. of argon. The aluminum powder produced was grey in color and was non-pyrophoric. The powder had a surface area of 10.1 square meters/gram and a calculated particle size of 0.24 micron.

Example 4

In this example two crucibles were positioned in the condensation zone of the vacuum tank in the manner described with respect to FIGURE 2. Nickel metal was placed in crucible 38 and aluminum metal placed in crucible 40. The crucibles were tilted towards each other to effect intermixing of the nickel and aluminum vapors to condensation on the cold plate 18.

The vacuum tank was closed and evacuated to a pressure on the order of 30–50 microns to remove most of the residual gases. During the evacuation the nickel and aluminum metal charges were heated to their respective melting temperatures. During this period, the pressure was adjusted by bleeding in argon. When the desired pressure of about 180 microns had been obtained, the nickel melt temperature was raised to about 1725° C., so as to cause evaporation of the nickel. Simultaneously, the aluminum melt temperature was raised to about 1250° C. so as to cause evaporation of the aluminum.

The rotating cooled plate 18 was positioned 6 inches above the metal vapor sources. The plate was cooled to a temperature of about 14° C. and was rotated at about 100 r.p.m.

The powder produced in this manner was pyrophoric and consisted of the alloy, Al_3Ni_2, aluminum and nickel.

It is apparent from the foregoing examples that the present process provides a relatively simple, economical process for the production of fine powders. Additionally, the present process lends itself equally to both small and large scale production of fine powders. For example, in large scale production, the metal can be continuously fed to the vapor source to provide for continuous operation.

Furthermore, since the present process permits condensation of the powder at a relatively short distance from the vapor source, the process can be carried out in a relatively confined space. This is of particular utility in the commercial production of fine powders.

The present process is also advantageous where it is desired to produce a powder having a particular surface area and "particle size" since such conditions as rate of removal of the powder from the condensation zone, the inert gas pressure, vapor source to condenser surface distance and accommodation coefficient can be widely varied to obtain the desired conditions.

While the specific embodiments of the invention have been described with respect to metals as the material for forming the powders, numerous other materials may be utilized. For example, any metal, metalloid, or compound which can be vaporized and deposited may be utilized.

Since certain changes may be made in the above process and apparatus without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. The process of producing fine metal powders comprising vaporizing a metal in an inert atmosphere at a pressure less than atmospheric to form a vapor stream which is directed into a condensation zone, passing a cooled surface through said condensation zone and collecting condensed metal vapors on said surface, removing the cooled surface, with the powder thereon, from said condensation zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the condensed powder from said surface, collecting the removed powder and again introducing said cooled surface into said condensation zone.

2. The process of producing fine metal powders comprising vaporizing a metal selected from the group consisting of aluminum, manganese, silver, chromium, beryllium, copper, boron, silicon, iron, nickel, zinc, mag-

nesium, titanium, zirconium, tantalum, tungsten, molybdenum, niobium, thorium and bismuth at a pressure less than atmospheric to form a vapor stream in a condensation zone of a vacuum chamber, passing a cooled surface through said zone, collecting the metal powder on said surface, removing the cooled surface, with the powder thereon, from said zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, and collecting the condensed powder.

3. The process of producing fine aluminum metal powders comprising vaporizing aluminum metal in an inert atmosphere to form a vapor stream which is directed into a condensation zone of a vacuum chamber, passing a cooled surface through said condensation zone, condensing the aluminum metal vapors on said surface, removing the cooled surface with the aluminum powder thereon from said condensation zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the aluminum powder from said surface and collecting the removed powder.

4. The process of controlling the particle size of metal powders comprising vaporizing a metal to form a vapor stream which is directed into a condensation zone of a vacuum chamber, passing the metal vapor stream through an inert gas, maintaining the inert gas at a substantially constant pressure value, condensing the metal vapors on a cooled surface, the particle size of the resultant powder being a function of said pressure value, said particle size increasing with decreasing pressure values and decreasing with increasing pressure values, rapidly removing the cooled surface, with the powder thereon, from the condensation zone to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the powder from said surface and collecting the removed powder.

5. The process of controlling the particle size of metal powders comprising vaporizing a metal selected from the group consisting of aluminum, manganese, silver, chromium, beryllium, copper, boron, silicon, iron, nickel, zinc, magnesium, titanium, zirconium, tantalum, tungsten, molybdenum, niobium, thorium and bismuth to form a vapor stream which is directed into a condensation zone, passing the metal vapor stream through an inert gas at a pressure of less than atmospheric, maintaining the inert gas at a substantially constant pressure value, condensing the metal vapors on a cooled surface in said condensation zone, the particle size of said powder being a function of said pressure value, said particle size increasing with decreasing pressure values, and decreasing with increasing pressure values, rapidly removing the cooled surface with the powder thereon from the condensation zone to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the powder from said surface and collecting the removed powder.

6. The process of producing fine aluminum metal powders comprising vaporizing aluminum metal in an inert atmosphere at a pressure less than atmospheric to form a vapor stream which is directed into a condensation zone, rotating a portion of a cooled surface through said condensation zone, condensing the aluminum metal vapors on said surface, rotating the cooled surface portion, with the aluminum powder thereon, from said condensation zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the aluminum powder from said surface, collecting the removed powder, and again rotating said cooled surface through said condensation zone.

7. The process of producing multi-metallic powders comprising vaporizing a plurality of metals to form vapor streams thereof which are directed into a condensation zone of a vacuum chamber, sequentially condensing the metal vapor streams on a moving cooled surface which passes through said zone, said condensing being carried out in an inert atmosphere at a pressure less than atmospheric, rapidly removing the cooled surface, with the powder thereon, from said condensation zone to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded to a collection zone, removing the powder from said surface and collecting the removed powder.

8. The process of producing alloy powders comprising simultaneously vaporizing the alloy metal constituents to form vapor streams which are directed into a condensation zone of a vacuum chamber, forming a mixture of the resultant metal vapors, condensing the mixture of metal vapors on a cooled surface, said condensing being carried out in an inert atmosphere at a pressure less than atmospheric, removing the cooled surface, with the alloy powder thereon, from said condensation zone to a collection zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, and collecting the powder.

9. In the process of producing fine powders wherein a material is vaporized and condensed, the improvement comprising vaporizing said material in an inert atmosphere to form a vapor stream which is directed into the condensation zone of a vacuum chamber, passing a cooled surface through said condensation zone, condensing said vapors on the cooled surface, removing the cooled surface, with the powder thereon, from said condensation zone at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the powder from said cooled surface and collecting the removed powder.

10. The process of producing fine metal powders comprising vaporizing a metal in an inert atmosphere at a pressure less than atmospheric in a vacuum chamber, passing a cooled surface through the resultant vapor metal stream, maintaining the accommodation coefficient of the cooled surface at approximately unity, condensing the metal vapor as a fine powder on said surface, removing the condensed powder from said vapor stream at a sufficiently fast rate to prevent heating of the condensed powder by the vapor stream whereby further crystal growth is impeded, removing the fine powder from said surface and collecting the removed powder in a non-oxidizing inert medium.

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