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PRODUCTION OF HIGH-PURITY, DUCTILE TITANIUM POWDER

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This invention pertains to the production of titanium powder, and more particularly to a high-purity, ductile titanium powder especially suited for use in powder metallurgy.

Most titanium metal is produced commercially by the chemical reduction of titanium chlorides with active reducing metals such as sodium and magnesium. The metal from such processes is referred to as sponge metal, and it is a relatively bulky, porous mass which must be purified, consolidated, and shaped. Most commercial procedures for consolidating and shaping the metal involve melting and solidification of the metal into ingots which are subsequently converted into a desired shape by such hot-working techniques as forging, rolling, and extrusion at temperatures ranging as high as 1000° C. At elevated temperatures, titanium is extremely reactive, particularly with such atmospheric substances as oxygen, nitrogen, and water vapor. Therefore, the melting and solidification of titanium is carried out in an inert atmosphere such as an argon atmosphere. An inert atmosphere is also used when titanium is heated for hot working, but in many instances, due to the size of the piece being worked and the nature of the particular hot-working operation, the actual working of the metal must be done in a normal atmosphere. As a result, large amounts of scale form on the titanium work piece, and this necessitates a subsequent descaling operation. Because of such difficulties, more practical methods for the fabrication of titanium have been sought. Cold working is a theoretical possibility, but titanium has not proved well suited to this method of fabrication because of its high strength, and its tendency to seize working surfaces and to work harden.

The fabrication of titanium articles by powder metallurgy avoids many of the difficulties and some of the steps encountered in producing and working titanium ingots. However, one of the reasons that powder fabrication has not replaced such techniques is the lack of a suitable process for the production of the metal powder. In many aspects, the grinding of titanium metal to a powder presents problems similar to those encountered in the fabrication of titanium ingots. The metal tends to seize and clog the grinding equipment. It loses ductility as a result of the grinding operation, and because of the high surface area inherent in a powder, it is extremely susceptible to deleterious contamination and a further loss of ductility. Moreover, there has also been the problem of finding a grinding action which produces a type of particle suited for powder metallurgy operations. It is particularly essential that the powder can be compressed to a porous compact having high green strength. The present invention is designed to overcome these difficulties in the preparation of metallurgical titanium powder.

The objects of this invention are accomplished by free impact milling, in an inert atmosphere at temperatures below 100° C., an anhydrous mixture of titanium and an alkalinous metal halide, the latter constituting at least 20% by weight of the mixture. The milling is conducted until substantially all the particles are in a size range which will pass at least a −14 mesh screen. Particles of this size and smaller are adaptable to powder metallurgy technique. The particles of titanium powder may be separated from the alkalinous metal halide by conventional means such as by dissolution of the halide in an aqueous medium.

The process of this invention employs free impact milling to produce the titanium metal powder. This type of milling action is imparted by a number of well-known mills which will be further discussed hereinafter. Briefly, free impact milling reduces particle size by placing the particle in free flight and pulverizing it by impact against the mill components or against other particles. In this invention, the milling of the titanium metal particles is conducted in the presence of an alkalinous metal salt, such as sodium chloride. It has been found that the salt overcomes seizing and the consequent build-up of titanium in the mill, a problem that previously presented much difficulty in milling titanium metal. It is also essential to the invention that the alkalinous metal halide be free of elemental alkalinous metal. If this mixture is to be obtained from the reduction of titanium halides with alkalinous metals, the absence of the free alkalinous metal can be insured by using an excess of titanium halide, thus causing the product mixture of titanium and salt to contain a small amount of titanium halide which will be in the form of the lower valent salts such as the trichloride and dichloride. The presence of these salts is evidenced by a greenish color in the mixture.

In a preferred embodiment of this invention, the anhydrous mixture of titanium and alkalinous metal halide is obtained from the chemical reduction of titanium chlorides with sodium, and it is devoid of unreacted sodium metal. Such a mixture is free impact milled at temperatures below 65° C. in an inert atmosphere until the titanium particles are in a size range between 30 and 325 mesh, and preferably with at least 50% below 60 mesh. Mesh sizes given throughout the specification are U.S. Standard sieve sizes.

Another embodiment involves the maintenance of ductility in the titanium powder. Although the grinding action in the presence of the salt is thought to be primarily one of separating and dispersing the agglomerates into crystallites, some work hardening of the titanium occurs. Consequently, for realization of ultimate ductility, the comminuted powder is heat treated to relieve the stress present due to working. Heating in the 400–750° C. range under protection from air and moisture will accomplish this objective. Protection from the air and moisture can be afforded in an inert atmosphere of argon or helium or the heating can be conducted in vacuum.

The titanium powder is separated from the salt either before or after the annealing heat treatment. The salt removal is preferably done by aqueous leaching, although when heat treated in presence of the salt above the melting point of the salt, a large part of the salt may be first separated by draining, decantation, filtering, etc.

For a clearer understanding of the invention, the following specific examples are given. These examples are intended to be merely illustrative of the invention and not in limitation thereof. Unless otherwise specified, all parts are by weight.

Example I

A crude titanium metal sponge was prepared by reacting titanium tetrachloride and metallic sodium in a mild steel reaction vessel. The purity of reactants and the protection from atmospheric contamination was such that the crude titanium was of good purity having a Brinell hardness of 100 as measured on a vacuum arc melted button. The reaction was adjusted so that substantially all the sodium metal was consumed and enough of titanium lower chlorides were present to be equivalent to about 0.5% of Ti in the salt. The reaction product was recovered as a solid cake. This cake was then broken up by hand to 3-inch lumps and passed through a crusher giving about $\frac{1}{2}$-inch lumps. These lumps were then reduced to about −6 mesh in a saw-toothed crusher in preparation for the production of the metal powder. The crushing steps were done as quickly as possible in air at 40% relative humidity and the granules sealed in cans. The metal during the crushing steps appeared to stay substantially encased within the salt so that only a very small fraction of metal surface was exposed to air. The free impact milling to metallurgical powder was done in a rotary sizing disintegrator having an eight-inch rotor mounted on a vertical shaft. The peripheral edges of the rotor traveled within $\frac{1}{32}$ inch of a 1" x 12 ga. bar screen enclosing the rotor section. This "scuff" screen carried a finer sizing screen fastened tightly around it; the latter screen had triangular holes of .012 in. inscribed diameter with about 90 holes per sq. in. The sizing screen was backed by another 1" bar screen and the whole screen assembly bolted together around the rotor. The mill was fitted with an air-tight casing so that the feed hopper, the rotor section, and product bins were interconnected for operation with the exclusion of air. The metal casing enclosing the milling chamber was cooled by contacting water coils. A thermocouple attached to the inner base of the rotor housing was used to indicate the mill temperature. A batch of the prepared granules was placed in the feed hopper and the casing closed, purged, and filled with argon. With the mill running at 3600 r.p.m. the feed was admitted and the feed rate controlled so that the temperature at the thermocouple did not exceed 100° C. Most of the time, it was between 50° C. and 65° C. The ground product was cooled in the argon and then removed to be leached in 5% aqueous nitric acid. The resulting titanium powder was dried and found to be 90% by weight −30 +325 mesh, and 75% of the −30 +325 mesh fraction was −60 mesh. This fraction was isolated and tested as a metallurgical powder. The microhardness of the particles was about 130 BHN, indicating that some work-hardening had occurred during the grinding operation. A portion of the powder was rolled into a green strip at about room temperature. Another portion of the powder was annealed in an argon atmosphere at 450° C. for 1 hr. The microhardness was reduced to 95 by the annealing. The annealed powder was also rolled to a green strip in the same manner as before, but a stronger strip was obtained. Either green strip could be rolled and otherwise conventionally handled through subsequent sintering and rolling steps to produce sound metal sheet.

Example II

A crystalline type of titanium metal was produced by reacting molten sodium chlorotitanite ($Na_{1.7}TiCl_4$) with a slight deficiency of molten sodium. The reaction mixture was allowed to digest at 850–1000° C. for about an hour to insure the complete consumption of the sodium. The mixture was then cooled and the reactor removed to a "dry room" where the relative humidity was held below 40%. The mass was removed from the reactor, broken into lumps, and reduced to granular sizes of from $\frac{1}{4}$ to $\frac{1}{16}$ inch in a rotary cutter. The granular product still handled in the low humidity was placed in the hopper of a cooled rotary screen-hammer mill with screen openings .03 inch in diameter. The air was displaced by dry argon. The feed was run through the cooled mill at a rate such that the product as discharged from the grinding zone was held between 50° C. and 75° C. The mill discharge was dropped into cold 5% nitric acid and the salt removed by washing and rinsing with water. The dried metal powder represented 95% of the titanium fed to the mill. Of this product, 100% passed a 14-mesh screen, 95% passed a 30-mesh, and 60% was under 60 mesh. The microhardness of the coarser particles was 95 BHN as compared with a sample of coarser particles isolated from the feed material which had a BHN of 85. A vacuum arc melted button made from a representative sample of the ground product screened to a +60 mesh powder had a Brinell hardness of 90. This powder was rolled to strip of satisfactory green strength. It could be extruded at moderate temperature to form a rod without previously annealing the powder. The −60 mesh fraction rolled to a stronger green strip, but the final product was somewhat harder.

Example III

Molten sodium chlorotitanite having a composition $Na_{1.9}TiCl_4$ was reduced with metallic sodium at a reaction temperature of about 825–850° C. The reactants were added alternately in 12 successive stoichiometric portions of about equal size. After each portion had reacted, about half the resulting sodium chloride was drawn out from the bottom of the reactor. After the twelve sequential reactions were completed, the mixture was held at 900–1000° C. for two hours to render the products free of unreacted sodium. The resulting mixture of titanium metal and sodium chloride was cooled, removed, crushed, ground, and leached as in Example II. The resulting metal powder was annealed by placing it in a column wherein it was fluidized for 15 minutes with argon preheated to 600° C. A screened portion of the annealed powder between 60 and 200 mesh, being about 70% of the whole product, was blended with a −200 mesh master alloy powder and rolled to a strong green strip sintered and re-rolled to form strong, .020 inch thick, alloy strip.

Example IV

Titanium tetrachloride was reduced to the metal with sodium using a 2% excess of $TiCl_4$. The reaction was carried out in a stainless steel reaction vessel at temperatures between 700° C. and 875° C. with the usual protection from atmospheric contamination. The reaction product was cooled in the vessel. The vessel was opened in an atmosphere where the relative humidity was between 20–30%, and the solidified mass, consisting of titanium, sodium chloride, and a small amount of titanium lower chloride, was removed from the vessel and broken into lumps and then into granules. The granular product was transferred to the feed hopper of a 12-inch diameter, heavy-duty Reitz Mill set to run at 3000 r.p.m. This mill had scuff and backing screens supporting a sizing screen punched with triangular holes 0.04 inch on a side. The discharge port of the mill was connected to a two-stage centrifugal separator. The first stage was designed to drop out titanium metal particles greater than 60 mesh. The second stage was designed to separate the fine metal from the salt. Provision was made to combine part of the salt with the coarse metal fraction. This mixture was recycled to the mill feed after passing it through an air-cooled heat exchanger, where its temperature was lowered in order to keep the mill discharge temperature below 65° C. The whole system, including the mill, was enclosed, and it was purged with argon prior to operation. The NaCl—Ti weight ratio in the crude mill feed was about 4–1. This ratio was also maintained in the recycled mixture of salt and the coarser titanium particles. At the end of the run, the ground mixture was leached in acidified water, washed, and dried. The screen analysis on the dried metal product was 92% minus 60 plus 200 mesh. This product was ductile, having a Brinell hardness of 90 when measured in an arc-melted button, and it was readily rolled into a compact of high green strength.

The material treated by the process of this invention consists essentially of ductile titanium and a halide salt of an alkalinous metal such as lithium, sodium, potassium, barium, calcium, and magnesium. Preferably the salt is that in which the titanium was formed by a reduction of a titanium halide. This mixture may be that obtained by the electrolysis of a titanium halide in a fused salt bath. Several such products or processes are described in the literature. The titanium may be made also by the chemical reduction of one or more of its halides by known methods in which the reducing agent is an alkalinous metal. The most common reducers are sodium and magnesium. The product obtained from the sodium reduction of a titanium chloride is the preferred material for use in this invention, particularly because of the substantially non-hydroscopic nature of the sodium chloride formed in association with the titanium permitting it to be handled without contamination in a normal atmosphere having a relative humidity below 40%. Magnesium may be used if one takes extra precaution to keep moisture away from the mixture at all stages of the process. The titanium should be pure enough to be inherently ductile, e.g., having a Brinell hardness number, measured on a vacuum arc-melted button, of less than 200, preferably below 110 such as in the 80–100 range. The salt associated with the mixture should be free of unreacted reducing metal. It has been found that even small amounts of reducing metal, for example, as little as .01% sodium, causes the charge to pack in the screen openings of a hammer mill, thus preventing normal discharge of the milled products. As a result, there is a deleterious temperature rise as the accumulated charge is subjected to excessive mechanical action by the rotor and eventually the mill becomes completely plugged. To avoid this trouble, the chemical reduction to produce the mill charge can be carried out with stoichiometric amounts of the reactants or, preferably, a slight excess of the titanium halide may be used to avoid the presence of unreacted reductant metal. For example, in the reduction of $TiCl_4$ with Na, a slight excess of $TiCl_4$ is used so that from 0.1 to 2.0% of titanium is left as lower chlorides after the completion of the reduction reaction. Such a procedure will result in a sodium-free metal-salt mixture. If the titanium is produced by chemical reduction, the whole reaction mixture may be used in the process of this invention, or, if desired, one may remove a portion of the salt from such a mixture. Partial removal of the salt is most easily done by draining the salt in the molten state during or just after the reduction. It is essential that the composition used in this invention contain at least 20% salt and preferably 40% or more by weight. Thus, one may well use the total product from the reaction $TiCl_4+4Na \rightarrow Ti+4NaCl$ which is 82.5% salt. Additional salt may also be present. The upper limit on the amount of salt is not particularly critical. However, excessive amounts of salt waste energy as unnecessary work is expended on the milling of the salt. While it is possible to make up the mill charge by adding salt to separated titanium metal sponge or crystals prior to milling, it is desirable to use a very intimate mixture of metal and salt, such as that obtained from the reduction of titanium chlorides with sodium. Both economy and quality of results make this reaction mixture preferred over one in which the salt and the metal are separately prepared. If the salt-metal mixture is in large pieces, it is broken to sizes suitable for mill feed. During such preparation for milling, precautions are taken to maintain the mixture in substantially anhydrous form. To do this, the mixture is handled in a substantially non-deliquescing atmosphere. If sodium chloride is the salt medium, it is less likely to pick up water from the air than such salts as $MgCl_2$ or $CaCl_2$. Consequently, the humidity of the contacting atmosphere in the work room is controlled accordingly. For sodium chloride, a relative humidity of not over 40% is adequate. A lower humidity is employed in handling the hygroscopic salts. Handling under an inert atmosphere during this preparation would, of course, achieve the desired protection. Also, it is well to avoid heating the mixture above about 100° C. during this preparatory stage.

Briefly stated, the preferred milling operation of this invention comprises subjecting the feed material to substantially dry, solid-state comminution in an enclosed, argon-purged, free edge-impact, classifying mill at temperatures below 65° C. Various types of mills may be used provided these essential conditions are achieved. Such mills may be described as having two active components, one of which imparts a velocity to the particles to be pulverized and throws them against a second component which in effect opposes the action of the first. The second component may be sationary or may travel in opposition to the first. Usually, one or both of these components act in a rotary fashion. The preferred mills are those in which the grinding action is that of free impact against an edge of the mill component. The edge is not necessarily sharp but seems to localize the force on the particle. These mills employ rotating cutters or bars as moving components. The stationary components may be opposing blades, bars, or bar screens, the latter being sometimes associated with a classifying screen or sieve. A considerable clearance, of the order of the diameter of the largest desired final particle size fraction or more, is maintained between the components. Thus, for producing −30 mesh powder the clearance in a hammer mill would be something like 1/16 or 1/32 inch. This avoids the undesired squeezing or shearing action which in general seems to cause seizing between the titanium particles themselves and between the particles and the mill components.

In metal grinding, the sharp mill edges usually become somewhat rounded, and such rounded edges are still satisfactory even when the radius of curvature is of the order of magnitude of the radius of the particle being milled. Clean operation is enhanced if the working edge leads the carrying structure with respect to the line or direction of impact. To provide this leading edge characteristic, the adjacent faces of the working parts are preferably cut-back or sloped away from the edge itself. Edges of stator parts of the mill against which particles are thrown may be similarly formed. The speed of travel of the moving edges is quite high. Some mills have peripheral speeds as high as 70,000 ft. per min. and may be used. The preferred edge velocity, however, lies in the range of from 700 to 25,000 ft. per min. Many mills suitable for this process are available. To illustrate some of them, reference is made to the 1950 edition of "Chemical Engineers Handbook," J. H. Perry, editor (McGraw-Hill Book Co.), (New York, Toronto, London), section 16, on Size Reduction and Enlargement, as follows:

Page 1140—The "Micro-Pulverizer"
Page 1141—The "Whirlwind Pulverizer"
Page 1141—The "Rietz Disintegrators"
Page 1142—The "Raymond Vertical Mill"
Page 1143—Disc attrition mills
Page 1130—Vibrating ball mills with angular grinding media

Some of these mills are primarily designed for grinding light material such as cereals, etc. in which case models of heavier and stronger structure may be specially made. The metal grinding task is relatively difficult, and it is preferred to use the rugged structure of the rigid rotary hammer fitted with bar screens as described in the examples.

The free impact milling of the titanium powder is done under protection of an argon atmosphere; hence, the mill must be enclosed or encased to maintain this protection. Such an enclosure can be built for almost any mill, it being simple to make enclosures for batch operation, but, if desired, feed and discharge locks can be supplied for semi-continuous or continuous operation. Absolute gas tightness is not essential, but usually the casing is assembled with close fits or gaskets and operated at a slight positive pressure so that inward diffusion of appreciable air or moisture is avoided. A vacuum-tight casing is, of course, useful in efficient purging. The mill and charge are purged with argon at the start and slow purging may be employed during operation.

Because of the high energy employed, cooling is necessary to keep the charge below 100° C. The cooling feature is not usually present in such mills but may be added in the form of cooling jackets, cooling coils attached to the housing, etc. Water cooling is of course a most efficient way of cooling the casing, but cooling may also be obtained with suitable external fins and circulated air. Another method of insuring good temperature control is to circulate argon through the mill and through a cooling heat exchanger. Pre-cooling the feed is effective. One of the best methods of cooling which is especially applicable to external closed circuit grinding is to recirculate a portion of the load through a heat exchanger returning the cooled coarse fraction to the mill, as illustrated in Example IV. The cooling rate and grinding load may be controlled in relation to each other to keep the desired temperature. Suitable temperature measuring devices are installed as near as possible to the region of greatest work for best control. Measurements taken on the material just leaving the grinding zone are generally satisfactory. A good operating temperature range is from 50° C. to 65° C. Lower temperatures are very satisfactory but economy of cooling and high production rates are best accomplished in the above temperature ranges. This temperature control is an essential part of the process. Excessive temperatures tend to cause packing of material and clogging of the mill. It is also believed that traces of moisture, and perhaps air, are present in the mill in spite of the precautions taken, and at temperatures above 100° C., these substances react with titanium and increase the hardness of the metal. The temperature limit applied here is in terms of average temperature of the material being ground. This is a practical way of stating the condition. Actually, it is believed that instantaneous temperature at the point of impact may be quite high. However, as long as the average temperature is maintained as specified, this high local temperature condition is so short-lived that degradation of the metal is not observed.

The classifying feature of the mill is preferably a screen closely associated with the pulverizing elements in order to simplify the maintenance of the protective atmosphere. However, mills using centrifugal means of sizing are useful. Even a system in which the classifier is separate from the actual grinding zone and the coarse fraction recycled to the feed may be used. Such recycling systems are preferably operated in the argon atmosphere, but the classifying may be done at room temperature in air and the coarse fraction mixed with the original feed. The classifying feature is controlled to give particles in the preferred broad size range of −30 +325 mesh. Particles finer than 325 mesh, while theoretically capable of producing high green strength in a compact, are easily contaminated due to the high specific surface. However, size ranges such as −60 +325 or −60 +200 mesh have satisfactory properties. In general, it is preferred that at least 50% by weight of the metallurgical powder produced is below 60 mesh. An external closed-circuit classifying system, as shown in Example IV, will provide a high yield of a desired mesh fraction with a minimum of fines. The recycling of material in this manner also affords a method of controlling the salt/Ti ratio in the grinding zone as well as provides a means of cooling. For example, if the salt/Ti ratio in the primary feed is not optimum for a particular operation, it can be corrected by using either a higher or lower ratio of salt to titanium in the recycled material.

The metal powder usually acquires some work-hardening during the grinding. To return the ductility of the powder to its ultimate or inherent value and thus improve its performance as a metallurgical powder, it may be annealed at temperatures between 400° C. and 800° C., preferably between 400° C. and 600° C. The annealing is carried out in a protective environment. For example, the metal-salt mixture in an anhydrous state still in the argon atmosphere as discharged from the mill may be heated and the powder later separated. It is preferred, however, to separate the powder by some means, such as aqueous washing and drying, prior to annealing. Heating in argon in an indirectly heated rotary kiln is one of the most practical methods for accomplishing this step. Fluidized bed heating with preheated dry argon is also suitable. Static heating in an oven or furnace does work but a long heating period is required, especially when large quantities are being handled. Care should be taken to have the powder dry before temperatures above 100° C. are reached. Drying and annealing after leaching may be accomplished in one operation, provided a suitable flow of protective gas is maintained to keep moisture away from the annealing zone. Vacuum drying prior to annealing is very satisfactory. Various annealing temperatures within the stated range may be used. However, it is desirable, in order to get maximum purity, to anneal at as low a temperature as possible. The degree of work hardening and the consequent need for annealing may vary considerably. When the feed metal is in the form of a friable mass of fine crystals, it is easily ground and very little work hardening occurs. In view of this, the annealing step is in many instances an optional step to obtain the highest possible ductility in the metal product.

The process of this invention is highly advantageous in that it will produce a high-grade ductile titanium powder of the correct particle size and ductility for powder metallurgy. Heretofore, there has been a need for a process which produces titanium metallurgical powder in practical quantities. The pure titanium produced by reduction reactions is recovered as sponge or coarse crystals which are unfit for direct use in powder compacting. The fine titanium produced as chemical precipitates or electrolytic deposits has been too impure to exhibit the necessary ductility. This invention, by virtue of the control of several critical conditions, provides the correct size in a metallurgical powder of high purity and ductility. In addition to these quality benefits, there are technical advantages within the process itself. One is the clean operation due to the absence of appreciable amounts of free reducing metal. It is known that moisture contaminates titanium; hence, it would seem essential that absolutely anhydrous ambient conditions be maintained. However, it has been shown that the Ti—NaCl mixture (which is devoid of free sodium) can be handled in an atmosphere of up to 40% relative humidity, provided the grinding temperature is held below 100° C. At these low temperatures, especially below 65° C., traces of moisture can be tolerated and, by exercising this temperature control, the costly maintenance of a completely dry room is avoided.

Since it is obvious that many changes and modifications can be made in the above-described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to said details except as set forth in the appended claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows.

I claim:

1. A process for producing a titanium powder suitable for use in powder metallurgy comprising free impact

milling in an inert atmosphere at temperatures below 100° C. a substantially anhydrous mixture of an alkalinous metal halide and titanium metal obtained from the reduction of titanium halides, said mixture being free of elemental alkalinous metal and having at least 20% by weight of alkalinous metal halide, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a 14-mesh screen.

2. The process of claim 1 wherein the temperature is below 65° C.

3. A process for producing a titanium powder suitable for use in powder metallurgy comprising free impact milling in an inert atmosphere and at temperatures below 100° C. a substantially anhydrous mixture of sodium chloride and titanium metal obtained from the reduction of titanium chlorides with sodium, said mixture being free of elemental sodium and having at least 20% by weight of sodium chloride, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a 14-mesh screen.

4. The process of claim 3 wherein the temperature is below 65° C.

5. The process of claim 4 wherein the mixture of sodium chloride and titanium metal is crushed and transferred to the free impact milling step in contact with air having a relative humidity of not greater than 40%.

6. A process for producing titanium powder suitable for use in powder metallurgy comprising free impact milling and classification in an inert atmosphere at temperatures below 100° C. of a substantially anhydrous mixture of sodium chloride and titanium metal obtained from the reduction of titanium chloride with sodium, said mixture being free of elemental sodium and having at least 20% by weight of sodium chloride, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a −60 mesh screen and are retained upon a 325 mesh screen.

7. The process of claim 6 wherein the temperature is below 65° C.

8. A process for producing a titanium powder suitable for use in powder metallurgy comprising free impact milling with a rotary sizing disintegrator in an inert atmosphere and at temperatures below 100° C. a substantially anhydrous mixture of an alkalinous metal halide and titanium metal obtained from the reduction of titanium halides, said mixture being free of elemental alkalinous metal and having at least 20% by weight of alkalinous metal halide, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a 14 mesh screen and at least 50% are −60 mesh.

9. The process of claim 8 wherein the temperature is below 65° C. and the anhydrous mixture is sodium chloride and titanium metal obtained from the reduction of titanium chlorides with sodium.

10. A process for producing a titanium powder suitable for use in powder metallurgy comprising free impact milling in in inert atmosphere and at temperatures below 100° C. a substantially anhydrous mixture of an alkalinous metal halide, titanium metal, and a small amount of titanium subhalide obtained from the reduction of titanium halides, said mixture being free of elemental alkalinous metal and having at least 20% by weight of alkalinous metal halide, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a 14-mesh screen.

11. The process of claim 10 wherein the temperature is below 65° C. and the anhydrous mixture is sodium chloride, titanium metal, and a small amount of titanium subchloride obtained from the reduction of titanium halides with sodium.

12. A process for producing a titanium powder suitable for use in powder metallurgy comprising free impact milling in an inert atmosphere and at temperatures below 100° C. a substantially anhydrous mixture of an alkalinous metal halide and titanium metal obtained from the reduction of titanium halides, said mixture being free of elemental alkalinous metal and having at least 20% by weight of alkalinous metal halide, said milling being conducted until the milled metal particles are in a size range wherein substantially all pass a 14-mesh screen, subsequently annealing the powder in a non-contaminating environment to temperatures in the range of 400–800° C.

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