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METAL PRODUCTION

William S. Wartel, Wilmington, Del., Roman J. Wasilewski, Coatesville, Pa., and Warren I. Pollock, Wilmington, Del., assignors to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

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This invention relates to the preparation of titanium base alloys. More particularly, it pertains to the production of ductile forms of these alloys by novel powder metallurgy techniques.

It is known that alloys of relatively soft metals, such as of copper and zinc, can be fabricated into simple or complicated shapes from their metal powders. Thus, particles of these metals in powdered form can be mixed in the desired proportions and compressed at room or elevated temperatures to a coherent compact. Upon sintering the mass (either during or after the compacting) dissolution of the alloying components results and a final working provides structures having physical properties similar to alloys from conventional melt-casting or fusion procedures.

When these techniques are applied to more refractory titanium and its alloys containing at least 50% by weight of that element, the relatively high strength characteristics of the titanium causes considerable difficulty in fabricating, i.e., rolling, extruding, forging, or drawing the sintered object. In addition, frequent conditioning and close control must be exercised over the working temperatures to control structure and avoid contamination. For successful working, elevated temperatures and of the order of about 900° C. to 1200° C. have been considered essential. Titanium at these temperatures is chemically very reactive toward moisture and with both oxygen and nitrogen. In addition, titanium in the pure state and at relatively low temperatures has a close-packed hexagonal structure known as the alpha phase which transforms at a temperature of about 885° C. to a body-centered cubic structure known as the beta phase. Conducting the working operation in the beta field is undesirable because of excessive grain growth and deep contamination from rapid diffusion of oxygen which ensues. In an effort to minimize these difficulties, the hot working operation is conducted with use of either (a) an atmosphere of relatively large volumes of an expensive inert gas, such as argon or helium, (b) a protective coating for the metal, or (c) without such inert gas and a consequent severe economic loss from oxide scale formation, and recourse to a subsequent treatment or series of treatments, such as scarfing, sand blasting, grinding, milling or pickling, designed to remove the scale and contamination encountered. None of these procedures has proved satisfactorily effective or economic in protecting the hot titanium metal from undesired oxygen and nitrogen contamination.

We have found that these and other difficulties in prior titanium-base alloy production can be effectively overcome, and one principal object of the invention is to provide novel and commercially useful methods for attaining these ends. Further objects are: to produce novel partially or completely homogenized titanium-base compositions or shapes and final products having physical properties substantially the full equivalent of or improved over alloys of the same composition produced from conventional melt-casting operations; to provide for the production of mill product forms of these alloys considered previously too difficult or impossible to prepare via powder metallurgy techniques; to provide for the production of ductile titanium base alloys readily amenable to economical shaping and fabrication; and to obtain these products

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from metal powder mixes which are compacted at relatively low pressures and temperatures, and without recourse to objectionable binder or lubricant use. Additional objects and advantages will be apparent from the ensuing description of our invention.

These objects are realized in this invention for obtaining our novel forms of titanium base alloy compositions. The invention comprises intimately associating at least 50% by weight of powdered, ductile alpha titanium with not more than an equal weight of a powdered, titanium-soluble alloying metal, forming the product mixture into a partially dense compact, heating said compact to partially homogenize it and produce a product with a density of at least 70%, an elongation of not less than 50% of that of a specimen of its unalloyed titanium matrix, similarly treated, a tensile strength of not more than 150% of that of said similarly treated unalloyed matrix, and which is capable of reduction in thickness under compression at room temperatures of not less than 35% without edge cracking, working the resulting partially homogenized product to 100% density under relatively low, not to exceed 600° C., temperature conditions and until not less than 40% reduction in cross-sectional area is obtained, and then heating the worked and reduced product to convert it to wholly homogeneous state.

In a more specific and preferred embodiment, the invention comprises production of a ductile titanium base alloy by mixing at least 60% by weight of pure, powdered, ductile titanium with at least one powdered, titanium-soluble alloying metal having a boiling point above 1700° C., pressing the resulting mixture into a partially dense, green compact, heating said compact to partially homogenize, bond or diffuse said alloying metal into the titanium matrix and to produce a product with an 85 to 95% density, an elongation value of not less than 75% and up to 85% of that of a specimen of the unalloyed titanium matrix similarly treated, a tensile strength of from 125% to 135% of that of a similarly treated specimen of the unalloyed titanium matrix, and which is capable of from 40% to 50% reduction in thickness under compression at substantially room temperature without edge cracking, working and reworking said partially homogenized product at temperatures ranging from about 25° C. to 200° C. to 100% density, and to give in excess of 75% and up to 90% reduction in cross-sectional area, and then heating the product obtained to temperatures ranging from 850° C. to 1500° C. until it converts to a wholly homogeneous alloy.

In practically adapting the invention to produce strong titanium base mill products containing, say, aluminum and one or more additional elements, such as vanadium, the ingredient metal powders or elemental powders and master alloy powders are blended in conventional rotating or tumbling type barrel or other forms of dry mixers. In such mixing, at least 50% by weight of substantially pure, powdered, ductile alpha titanium metal is associated with not to exceed an equal weight of the relatively pure, powdered alloying metal or mixtures of such metal or its alloys and such amounts are used as will provide a final composition which on chemical analysis will correspond, by weight to from 50% to 99% and preferably from 60% to 95% of titanium, and from about 1% to not to exceed 50% and preferably from 5% to 40% of the alloying metal or mixtures. Alloying metals useful herein comprise those having a boiling point above 1700° C. and which are soluble at elevated temperatures in the titanium. Examples thereof include aluminum, iron, chromium, molybdenum, vanadium, niobium, etc. The titanium used should have a Brinell hardness number (BHN) of not to exceed about 200 and preferably such value is below 100 to facilitate the relatively low temperatures of working undertaken in the invention. To insure final

product uniformity, the metal components should be of suitable particle size and distribution, e.g., 100% of the particles should pass a -10 mesh screen and, if desired, up to 10% or more of such particles can pass 325 mesh screen.

Upon attaining intimate blending or mixing of the particles, the powder mixture is consolidated to a coherent compact, billet, sheet or strip in conventional die molding, rolling or pressing equipment designed to shape the mixture into a coherent compact capable of withstanding the subsequent handling and working treatments to which the compact will be subjected in the later stages of the process. For example, the compaction can be effected by charging the powder mixture to a suitable mechanical or hydrostatic press, or to die or molding equipment in which sufficient pressure can be exerted to form the powders into the desired billet or compact. Pressures utilized in the compaction can vary and range from, say, a lower limit of about 10 tons per square inch (t.s.i.) and up to as high as 75 or 100 t.s.i. depending upon the particular form of pressing operation undertaken, the capacity of the powders to form a coherent mass and the green strength and density desired in the preformed compact.

The compact produced in the pressing operation is then partially homogenized by direct heating to bond and partially diffuse the alloying metal or metals into the titanium matrix, as opposed to the essentially full alloying accomplished in the sintering step of prior conventional powder metallurgy procedures. This heating is carried out in conventional vacuum or inert atmosphere furnacing means with the temperature and time of treatment being so controlled that the degree of homogenization obtained will be such that only the matrix in the immediate vicinity of the alloying metal or metals will have acquired a greater degree of toughness than that exhibited by the original, pure base titanium metal while the main body of said matrix will have retained its original ductility. Furthermore, the strength of the bonding under these conditions will be such that, during subsequent working of the partially homogenized product, even highly-brittle master alloy particles present in the matrix are deformed and elongated very markedly in the working direction without losing cohesion with the matrix. When the proper amount of such bonding is achieved according to this invention by partial homogenization, the piece will stand at least 35% thickness reduction in compression without edge cracking. Excessive homogenization markedly increases strength and reduces workability so that the necessary amount of cold working is no longer feasible.

The temperature and the duration of treatment required to effect the partial homogenization necessary can be varied. Generally, such temperature can range from about 900° C. to 1500° C. while temperatures ranging from about 1000° C. to 1300° C. are preferred to achieve optimum results. The time of treatment within the preferred temperature range usually ranges from about 1 minute to 30 minutes and will depend upon the particular temperatures used and the nature of the composition being treated. Preferably, the heating is carried out under vacuum, and, if desired, it can be effected under an atmosphere of an inert rare gas, such as argon or helium. The homogenization treatment is continued until partial bonding or diffusion of alloying metal with the titanium metal takes place and a product having a density of at least 70% and preferably from 85% to 95% results, said product having an elongation not less than 50% and preferably not less than 75% to 85% of that of a similarly treated specimen of the unalloyed titanium matrix metal, a tensile strength of not more than 150% and preferably not to exceed from 125% to 135% of such similarly treated matrix, and capable of reduction in thickness under compression at room temperature to not less than 35% and preferably to from 40 to 50% without edge cracking.

When desired partial homogenization has been achieved, the partially alloyed product is worked at relatively low

temperatures (and, if necessary, further heat treated to relieve internal stresses and reworked) until a fully (100%) dense form of mill product or shape results. In such working, temperatures not exceeding 600° C. and preferably, ranging from about 25° C. to 200° C., are utilized, and recourse is had to such conventional type procedures as extrusion, forging, swaging, pressing or rolling or any desired combination of these treatments.

The cold worked, 100% dense, partially homogenized mill product obtained from the compression working operation is then converted to a completely homogeneous alloy possessing desired ultimate tensile strength, yield strength, reduction in area and elongation characteristics by direct heat treatment in furnacing means of the type above mentioned at 900° C. to 1500° C. temperatures and under vacuum or in an atmosphere of an inert gas. As a result, billets, rods, tubes, sheets, plates, strips, foils, wires and the like, are produced which are adapted for fabrication, if desired, into other forms of useful structural materials.

To a clearer understanding of the invention, the following specific examples are given in which parts and percentages mentioned are by weight. These are merely illustrative and are not to be taken as limiting the underlying principles and scope of the invention.

EXAMPLE I

A titanium base powder mixture consisting of 85% of -60 mesh titanium powder and 15% of -270 mesh, 60% aluminum-40% vanadium master alloy, was prepared by blending these powders at room temperature in a conventional-type blender apparatus. The mixture obtained was then rolled directly to 0.020" green strip in a single pass. This green strip was then heated for 15 minutes at 1200° C. to partially homogenize it, after which it was cooled to room temperature and cold rolled at 25° C. to 0.010" without edge cracking. The resulting sheet material was then annealed at 1030° C. for one-half hour to complete homogenization after which the strip was finally cold-rolled at 25° C. to 0.003". The fully dense foil obtained was then heat treated for one hour at 900° C., aged at 600° C. for another hour and then furnace cooled to provide a useful foil product which had a tensile strength of 180,000 p.s.i. and an elongation of 5%. On chemical analysis it was found to consist of an alloy of 8.85% aluminum, 6.3% vanadium, balance titanium. A portion of this foil upon being heat treated at 900° C. for one hour, furnace cooled, and tested in tension had a tensile strength of 150,000 p.s.i., a ductility in elongation of 12.5%, and a bend radius of less than 1T. Its exceptional ductility in tension and bend characteristics are by-products of its ability to work cold, and reflects the low interstitial content of the finished foil.

EXAMPLE II

Using a conventional type cone blender, 100 parts of a master alloy powder comprising 60% aluminum and 40% vanadium, were mixed at room temperature with 900 parts of titanium powder of -60 mesh particle size. The mixed powders were then pressed at a pressure of 12.5 t.s.i. into two slabs of approximately 5" x 5" x 0.35" dimension having densities of 72-74% of theoretical.

Both green compacts were then partially homogenized by heating in vacuo at a temperature of 1025° C. for 20 minutes after which they were furnace cooled to room temperature. Examination of the micro structure of the metal compacts after this sintering treatment showed that alloying had taken place to a limited extent. The density of the slabs after the sintering was 77-78% of theoretical.

The high ductility, as reflected by elongation, and low strength of the partly homogenized product (of importance in permitting relatively easy fabrication), and other advantageous physical properties of the metal during the working stages of this process, are shown in the following table.

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Table I

	Ultimate Tensile Strength	Yield Strength, 0.2 Offset	Elongation, Percent (in 1" gauge)
1 hr., 900° C. water quenched.....	114,000	88,000	15
Same alloy wrought from ingot.....	140,000	130,000	10

Both slabs were then cold-rolled at 25° C. to 45–65% reduction in cross section area without edge cracking to obtain a product having a density of 95–97% of theoretical, and each was then vacuum annealed for 30 minutes at 1065° C., and again furnace cooled and cold-rolled to a final thickness of 0.030". Further examination of the micro structure revealed the existence of very limited alloying with no residual porosity being observed.

Final homogenization of the alloy was then brought about by heating the strip in vacuo for four hours at 1100° C. followed by furnace cooling. After this final heat treatment the strip consisted of a fully alloyed product, this being evidenced by its structure and physical properties. In respect to such properties as hardness, strength, and elongation, the product was indistinguishable from an alloy of the same composition prepared from conventional casting and working of the ingot to render the product fabricable on rolling into a sheet material useful for obtaining structural products.

EXAMPLE III

A metal powder mixture comprising 90 parts of titanium passing a 60 mesh screen, and 10 parts of master alloy powder of 60% aluminum and 40% vanadium, was prepared by blending the powders in a mixer in the proportions mentioned at room temperature. This mixture was then rolled directly in a conventional mill to 0.011"–0.012" sheet. The resulting sheet was then partially homogenized by heating in vacuo at 1000° C. in a resistance furnace for 15 minutes and furnace cooled to provide a product with a density of 85% of theoretical. The partially homogenized sheet was then cold rolled at 25° C. to 0.002" and was heat-treated at 1010° C. for one-half hour. After furnace cooling, the resulting sheet was further rolled at 25° C. to 0.001" and was finally annealed in vacuum at 850° C. for one-half hour. The foil resulting from this treatment was found to be equivalent in microstructure and hardness to a conventional product of similar composition. Its bend radius was determined to be 1T which is significantly better than the 5T value obtained for foil made via conventional melting route.

EXAMPLE IV

A blend of 90% titanium powder (–30 mesh) and 10% of 60% aluminum–40% vanadium master alloy powder (–200 mesh) was prepared by mixing the powders at room temperature in a conventional blender apparatus. The mixture obtained was formed into a billet 2" in diameter and 4" long by hydrostatically pressing it at room temperature at 25 t.s.i. On heating this billet at 1065° C. in vacuo for 15 minutes and furnace cooling it, partial homogenization resulted. The billet was then heated to 600° C. in argon and extruded at a ratio of 4:1 to a round rod. The extruded rod was then fully homogenized by heat-treating it for one hour at 1200° C. in vacuo and in a resistance furnace and was furnace cooled. The product alloy was found to be comparable in all respects to a product of the same composition obtained from a conventional melting and casting operation.

EXAMPLE V

A slab of dimensions 5" x 5" x 1" was prepared by pressing at 12 t.s.i., a powder mixture consisting of 10% master alloy powder made up of 60% aluminum–40% vanadium and of –200 mesh particle size, with 90% of titanium powder of –60 mesh particle size. The density

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of the green bar thus obtained was 73% of theoretical. This bar was then partially homogenized by heating it in an induction furnace for 30 minutes at 1030° C. in a vacuum, and the partially homogenized product was then sawed into bars 1" x 1" x 5" which were cold-forged at 25° C., using open flat dies to approximately 0.60" square by 10" long. No edge cracking was observed in the product. For the forging operations pressures from an initial breakdown pressure of 20 t.s.i. to a final pressure of about 60 t.s.i. were used. After the forging treatment the bars were found to be essentially fully (100%) dense.

The bars were then heated for 30 minutes at 1000° C. in a vacuum in a tube resistance furnace and were furnace cooled. Two of the bars were then cold-rolled (at 25° C.) and two were warm-rolled (600° C.) to 0.300" diameter round rods. After a further heat treatment for 15 minutes at 1200° C. in vacuum, they were then cooled and found fully homogenized and 100% dense.

EXAMPLE VI

A 1000 part mixture consisting of 13% V, 11% Cr, 3% Al balance Ti was prepared by blending at room temperature Ti and V–Cr–Al master alloy powders in a twin-shell cone blender. 730 parts of –60 +200 mesh, 100 BHN, Ti was blended with 270 parts of –20 +325 mesh master alloy. The mix was roll compacted on a 4" diameter 2-high rolling mill to 85% dense green strip, 3½" wide and 0.027" thick. The strip was partially homogenized in an argon atmosphere by induction heating the strip to 1300° C. for 15 minutes. An increase in tensile strength of approximately 20% over that of the pure Ti used accompanied this step. The density increased to 88–90% of theoretical. The strip was then cold rolled at 25° C. on the 2-high mill to 0.019 to 0.020". A stress relief heat treatment at 650° C. for 15 minutes was applied. The strip was cold rolled at 25° C. on a 4-high mill to 0.010 and fully homogenized at 1300° C. for 15 minutes. Metallographic examination showed a single phase, all beta structure. The tensile data obtained on the 0.010" sheet were: UTS—140,000 p.s.i.; YS—135,000 p.s.i.; percent E (1") 20%. These values are equivalent to data reported on annealed conventional sheet product. The 0.010" gage material was useful as face sheet in honeycomb panels for aircraft construction.

EXAMPLE VII

A blend consisting of 282 parts of –60 mesh titanium powder, 6 parts of –60 mesh chromium, and 6 parts each of –100 mesh iron and molybdenum powders was prepared in a conventional mixer apparatus. This blend was then conventionally rolled at room temperature directly to green strip 0.008"–0.010" thick. The resulting sheet was then partially homogenized at 1200° C. for 15 minutes under an atmosphere of helium. Its density was then determined to be about 90%, and a photomicrograph showed only partial alloying to exist. The sintered sheet was again cold-rolled to full density at room temperature to 0.005"–0.007", heated again at 1200° C. for 15 minutes in helium, furnace-cooled and was finally room temperature cold rolled to 0.001" and stress relieving at 450° C. for 1 hour. The final foil obtained was equivalent in microstructure and hardness to a conventional alloy of the same composition.

A bend radius of 1T was obtained on this foil, which was significantly better than the 5T value obtained on a foil of the same composition but produced via the conventional melting route.

While described as applied to certain specific embodiments, the invention is not restricted thereto. Our novel alloys comprise homogeneous powder metallurgy compositions and shaped objects in mill product or billet form adapted for further fabrication in conventional metal working techniques, e.g., forging, rolling, extrusion and drawing. They contain, on a weight basis, as essential ingredients at least 50%, and preferably from 60% to

95%, of pure titanium, together with not more than 50%, and preferably from 5% to 40% of an alloying metal or mixtures of such metals. Trace amounts, not to exceed a total of about .5% of carbon, nitrogen, or oxygen impurities also can be present. The alloying metal is added or incorporated in the titanium in either the elemental state, as mutual alloys or mixtures, or as a master alloy with titanium, and the sum of their amounts generally ranges from .5% to 50%, by weight. Preferably, when relatively low melting alloying metal are used, alloying with the titanium to form master alloys having higher melting points is resorted to.

Alloying metals most useful comprise those relatively low in volatility and which boil above 1700° C. at atmospheric pressure and are soluble in the titanium at temperatures above its transition temperature in the pure state. Such temperatures range from about 885° C. to 1600° C., and the alloying metals can be soluble in the titanium between such transition temperature and the lowest melting point of the titanium base composition being formed. Particularly useful alloying metals, and in the amounts mentioned comprise beryllium, aluminum, tin, copper, vanadium, chromium, manganese, iron, cobalt, nickel, zirconium, niobium, molybdenum, tantalum, tungsten, hafnium and rhenium. Thorium, palladium, platinum, cerium and lanthanum are also utilizable and preferably in amounts ranging from about .5 to 10% on a weight basis.

As indicated in the examples, the invention is advantageously useful in alloying titanium with greater than 5% amounts and, if desired, with from 8% to 12% by weight of aluminum as a single additive or in conjunction with another added metal. Heretofore, the use of these amounts of aluminum has been considered disadvantageous and has been avoided because as increasing amounts of aluminum are added difficulties in fabricating the alloy increase. Thus, according to McQuillan and McQuillan, in *Titanium: Metallurgy of the Rarer Metals*: No. 4 (1956), aluminum additions to titanium in amounts greater than 4% while affording rapid increase in the tensile strength and yield strength of the binary alloys resulted in poor workability and decreased the amount of cold rolling to which the alloy could be subjected. Furthermore, the recrystallization temperature which for pure titanium is 600° C. becomes elevated by aluminum addition and at the 5% addition level is already 800° C. In an effort to counteract this poor workability, small amounts of various beta stabilizers or eutectoid formers which tend to widen the alpha-beta field and lower the yield strength without affecting the ultimate strength, are added so that working of the alloy becomes favorable. However, even with the addition of these agents the use of aluminum in amounts above about 8% in a titanium base alloy has not been feasible without encountering the disadvantages mentioned and a resulting extreme brittleness which makes working of the alloy, either hot or cold, virtually impossible. By this invention a two-phase alloy consisting of a ductile aluminum-poor matrix with a uniformly distributed-aluminum-rich phase (either of these with one or more additional alloying elements) becomes readily fabricable even if the overall aluminum content should exceed 8%.

As noted above, the required degree of alloying will be controlled by a choice of time and temperature of the initial partial homogenization step, and the subsequent intermediate heat-treating steps, as well as by the choice of alloy or master alloy present in the composition under treatment, and the particle sizes of the powders used. We have found that certain powder characteristics advantageously assist in successfully carrying out the various steps of the process, and insure in the product alloy final mechanical properties at least equivalent to the corresponding alloy wrought from ingot. In contrast to conventional powder metallurgy practices which, in general, require fractions of powder finer than 200 mesh, use of

relatively coarse titanium base metal powder is preferred in the fabrication of the ductile alloys of this invention. Thus, excellent results can accrue when such metal powders are of predominantly -30 to +200 mesh particle size, with their surface areas ranging between 0.01 and 0.10 m.²/g. and, for the most part, about 0.03 m.²/g. Advantageously, the use of such low surface-area powders minimizes contamination of the alloy product as a result of nitrogen and oxygen being absorbed on the surfaces of the powders. These two interstitial contaminants degrade the properties, especially the ductility of the alloy. Although it has been found desirable to use relatively coarse particle-size fractions of the titanium base powders, we prefer to use with these relatively fine fractions alloying metal or master alloy powders in the size range of, say -200 to +325 mesh.

While the hardness characteristics of the powdered alloying metal particles used will have some effect on the ultimate product, this is not a critical aspect of the invention. It is essential that the titanium base material be in quite pure state to insure necessary ductility, an essential property for titanium usefulness. Titanium with the Brinell hardness values mentioned, i.e., under 200 and preferably near 100 or below, is most usefully employable. The Brinell hardness numbers given were determined in accordance with the method described in the 1955 Book of Standards of the American Society for Testing Materials, part 1, page 1573, under ASTM designation E10-54T. If desired, use can be made of ductile forms of titanium which may contain small amounts (say 2-3%) of an alloying metal such as vanadium, zirconium or aluminum, the presence of which will not adversely affect titanium ductility or raise its hardness value beyond those specified.

The cold working treatment contemplated is important in procuring optimum results in the invention. The degree of cold working to which the partially homogenized alloy can be subjected can be determined from comparing the properties of the pure matrix material and the alloy material being fabricated. For purposes of such comparison and definition, two powder compacts as follows are prepared: A powder mix made up of 90% by weight titanium powder (-60 to +200 mesh particle size) and 10% of a master alloy powder mix comprising 60% aluminum and 40% vanadium. A rod of 5/8" diameter was formed by compacting this powder mix hydrostatically at 30 t.s.i. Using a portion of the same powdered titanium, a similar pure metal compact of the same dimensions was prepared using the same pressure. Both bars were then heated in vacuo for 30 minutes at 1025° C. Tensile test specimens 1" in length and compression test discs 1" high were machined from the heat-treated, 89% dense, bars. On testing these specimens, the following results were obtained:

	Pure Ti Specimen	Ti, 6Al-4V Specimen
Yield, 0.2% offset (p.s.i.)	48,000	56,000
Ultimate Tensile Strength (p.s.i.)	55,000	60,000
Percent Elongation	20.0	18.0
Edge Cracking first occurred when reduction under compression was.	62% at 70 t.s.i.	51% at 78 t.s.i.

In fabrication of alloys in accordance with the invention, the first heating after the compaction affects the mechanical and deformation properties of the matrix. To obtain optimum results, the properties of the alloy being fabricated can be defined relative to the properties which would be exhibited by a similarly treated compact of the unalloyed matrix material, as illustrated by the foregoing testing of the two described compacts. We have found that working within the following limits affords production of excellent alloy mill products: The partially homogenized alloy compact in respect to elongation should exhibit not less than 50% and preferably

should be from 75 to 85% of that of its unalloyed, similarly treated titanium matrix; the tensile strength of the heat-treated alloy compact should show not more than 150%, and preferably not more than 125-135% of that of the unalloyed matrix material; the heat-treated alloy compact should show a reduction in thickness value under compression at room temperature of not less than 35% and preferably from 40-50% reduction without edge cracking.

Depending on the particular composition of the cold formed mill product, conversion thereof to a metallurgically homogeneous alloy having desired high structural strength is brought about through simple direct heat treatment. The extent of heating necessary to cause complete solution or homogenization of the added alloying agents in the titanium generally ranges from the transition temperature of the titanium component and the lowest melting point of the metal composition, i.e., from about 850° C. to about 1600° C. depending on the melting point of the compositions being treated, the purity of the titanium component and/or the type of product or finish desired. Preferably, temperatures of the order of from about 1000 to 1300° C. are employed. The heat treatment is continued until complete solution of the added alloying material in the titanium results.

As already noted, comparative photomicrographs of specimen products of this invention made at various stages of their production with prior art products will demonstrate their distinguishing characteristics and establish that the completely homogenized structures of our invention are identical with alloys of the same composition prepared from conventional melting and casting methods.

We claim as our invention:

1. A method for producing a homogeneous titanium base alloy composition, comprising pressing into a coherent, partially dense compact an intimate powder mixture of at least 50% by weight of ductile titanium and not more than an equal weight of at least one alloying metal having a boiling point above 1700° C., which is soluble in pure titanium at temperatures above its transition temperature, partially homogenizing said compact through heating at temperatures ranging from about 900-1500° C. for from about 1-30 minutes until partial bonding of the alloying metal with the titanium takes place and to form a product having at least 70% density, an elongation of not less than 50% of that of a specimen of its unalloyed titanium matrix similarly treated, and a tensile strength of not more than 150% of said similarly treated matrix, said product being capable of reduction in thickness under compression at room temperature to at least 35% without edge cracking, subjecting the partially homogenized product to working at a temperature below 600° C. and until not less than 40% reduction in cross-sectional area is obtained, and then heat treating the resulting product at temperatures ranging from about 850° C.-1600° C. to convert it to a metallurgically homogeneous alloy.

2. A method for producing a homogeneous titanium base alloy composition comprising pressing into a coherent, partially dense compact an intimate powder mixture of at least 60% by weight of ductile titanium and an alloying metal having a boiling point above 1700° C. which is soluble in pure titanium at temperatures above its transition temperature, heating said compact at temperatures ranging from about 900° C.-1500° C. for from 1-30 minutes until partial bonding of the alloying metal with the titanium metal takes place and partial homogenization thereof and formation is obtained of a product having an 85-95% density, an elongation of from 75-85% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of from 125-135% of said similarly treated matrix, said product being capable of reduction in thickness under compression at room temperature to from 40-50% without edge

cracking, subjecting the partially homogenized product to working at temperatures ranging from room to 600° C. until reduction in cross-sectional area in excess of 75 and up to 90% results, and then heating the resulting product at temperatures ranging from about 850° C.-1600° C. to convert it to a metallurgically homogeneous alloy.

3. A method for producing a homogeneous titanium base alloy composition, comprising pressing into a coherent, partially dense compact an intimate powder mixture of at least 50% by weight of ductile titanium and not more than an equal weight of at least one alloying metal which is soluble in the titanium at temperatures above its transition temperature and has a boiling point above 1700° C., heating said compact at temperatures ranging from about 900° C.-1500° C. for from 1-30 minutes until partial bonding of the alloying metal with the titanium metal takes place and partial homogenization thereof and formation is obtained of a product having a density of at least 70% density, an elongation of not less than 50% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of not more than 150% of that of said similarly treated titanium matrix, said product being capable of reduction in thickness under compression at room temperature to not less than 35% without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 600° C. to not less than 40% reduction in cross-sectional area, and then converting the product obtained to a metallurgically homogeneous alloy by heat treatment at temperatures ranging from about 850° C. to 1600° C.

4. A method for producing a homogeneous titanium base alloy composition, comprising pressing into a coherent, partially dense compact an intimate powder mixture of at least 60% by weight of ductile titanium and a balance of at least one alloying metal which is soluble in the titanium at temperatures above its transition temperature and has a boiling point above 1700° C., partially homogenizing said compact by heating it at temperatures ranging from about 900° C.-1500° C. for 1-30 minutes until partial bonding of the alloying metal with the titanium metal takes place and the product has a density of at least 70%, an elongation of not less than 50% of that of a specimen of its similarly treated unalloyed titanium matrix, and a tensile strength of not more than 150% of that of said unalloyed similarly treated titanium matrix, said product being capable of reduction in thickness under compression at room temperature to not less than 35% without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. to not less than 40% reduction in cross-sectional area, and then converting the resulting, consolidated product to a metallurgically homogeneous alloy by heat treatment at temperatures ranging from about 850° C. to 1600° C.

5. A method for producing a titanium base metal alloy composition, comprising pressing into a coherent compact an intimate powder mixture of at least 50% and up to 99% by weight of ductile titanium and from about .5% to not to exceed 50% by weight of at least one alloying metal having a boiling point above 1700° C., which is soluble in pure titanium at temperatures above its transition temperature, partially homogenizing said compact by heating at a temperature ranging from about 900° C.-1500° C. for from 1-30 minutes until partial bonding takes place between the alloying metal and the titanium and a product is obtained having a density of at least 70%, an elongation value of not less than 50% of that of a similarly treated specimen of its unalloyed titanium matrix and a tensile strength of not more than 150% of that of said similarly treated titanium matrix, said product being capable of at least 35% reduction in thickness under compression at room temperature without edge cracking, working the partially

homogenized product while maintaining the same under temperatures ranging from about 25° C. to not to exceed 600° C. and to not less than 40% reduction in cross-sectional area and then converting the resulting product through heat treatment at temperatures ranging from about 850° C.-1600° C. to a metallurgically homogeneous alloy.

6. A method for producing a titanium base metal alloy which comprises pressing into a coherent compact an intimate powder mixture made up of at least 60 to 95% by weight of ductile titanium and from about 5% to about 40% by weight of at least one added alloying metal which is soluble in the titanium at temperatures above its transition temperature and has a boiling point above 1700° C., partially homogenizing said compact by heating it at temperatures ranging from about 900° C.-1500° C. for 1-30 minutes until there is obtained a product with a density of from 85-95%, an elongation value of not less than 75% to 85% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of from 125-135% of that of said similarly treated titanium matrix, said product being capable of at least 40-50% reduction in thickness under compression at room temperature without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. until a shaped product reduced in excess of 75% and up to 90% in cross-sectional area results, and then heating the consolidated product at temperatures ranging from 900-1500° C. until it becomes converted to a fully homogenized alloy.

7. A method for producing a titanium base metal alloy which comprises pressing into a coherent compact an intimate powder mixture made up of at least 60 to 95% by weight of ductile titanium and from about 5% to about 40% by weight of added aluminum, heating the preformed compact obtained at a temperature ranging from about 1000° C.-1300° C. for 1-30 minutes until partial homogenization and bonding of the alloying metal with the titanium metal takes place and formation results of a product with a density of from 85-95%, an elongation value of not less than 75% to 85% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of from 125-135% of that of said similarly treated titanium matrix, said product being capable of at least 40-50% reduction in thickness under compression at room temperature without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. until a shaped product reduced to in excess of 75% and up to 90% in cross-sectional area results, and then heating the consolidated product at temperatures ranging from 900-1500° C. until it becomes converted to a fully homogenized alloy.

8. A method for producing a titanium base metal alloy which comprises pressing into a coherent compact an intimate powder mixture made up of at least 60 to 95% by weight of ductile titanium and from about 5% to about 40% by weight of added aluminum and vanadium, partially homogenizing the preformed compact obtained by heating it at a temperature ranging from about 1000° C.-1300° C. for 1-30 minutes until partial bonding of the titanium with said aluminum and vanadium takes place and a product results having a density of from 85-95%, an elongation value of not less than 75% to 85% of that of a similarly treated specimen of its unalloyed

titanium matrix, and a tensile strength of from 125%-135% of that of said similarly treated titanium matrix, said product being capable of at least 40-50% reduction in thickness under compression at room temperature without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. until a shaped product having in excess of 75% and up to 90% reduction in cross-sectional area results, and then heating the consolidated product at temperatures ranging from 900-1500° C. until it becomes converted to a fully homogenized alloy.

9. A method for producing a titanium base metal alloy which comprises pressing into a coherent compact an intimate powder mixture made up of at least 60 to 95% by weight of ductile titanium and from about 5% to about 40% by weight of added aluminum, chromium and vanadium, partially homogenizing the preformed compact obtained by heating it at a temperature ranging from about 1000° C.-1300° C. for 1-30 minutes until bonding of the titanium and aluminum, chromium and vanadium takes place and a product results with a density of from 85-95%, an elongation value of not less than 75% to 85% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of from 125-135% of that of said similarly treated titanium matrix, said product being capable of at least 40-50% reduction in thickness under compression at room temperature without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. until a shaped product having in excess of 75% and up to 90% reduction in cross-sectional area results, and then heating the consolidated product at temperatures ranging from 900-1500° C. until it becomes converted to a fully homogenized alloy.

10. A method for producing a titanium base metal alloy which comprises pressing into a coherent compact an intimate powder mixture made up of at least 60 to 95% by weight of ductile titanium and from about 5% to about 40% by weight of added chromium, iron and molybdenum, partially homogenizing the resulting preformed compact by heating it at a temperature ranging from about 1000° C.-1300° C. for 1-30 minutes and until partial bonding of the alloying metals with the titanium takes place and a product is formed having a density of from 85 to 95%, an elongation value of not less than 75% to 85% of that of a similarly treated specimen of its unalloyed titanium matrix, and a tensile strength of from 125-135% of that of said similarly treated titanium matrix, said product being capable of at least 40-50% reduction in thickness under compression at room temperature without edge cracking, working the partially homogenized product at temperatures ranging from about 25° C. to 200° C. until a shaped product having in excess of 75% and up to 90% reduction in cross-sectional area results, and then heating the consolidated product at temperatures ranging from 900-1500° C. until it becomes converted to a fully homogenized alloy.

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