

[54] **METHOD OF HOT COMPACTING
TITANIUM POWDER**

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[58] Field of Search **29/420.5, 420, DIG. 31, 29/DIG. 45; 75/226**

[56]

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[57]

ABSTRACT

Hot isostatic compacting of titanium or titanium-base alloy powder to a product having controlled interstitials and microstructure and possessing outstanding end properties, low porosity and high density and one which does not require subsequent forging.

15 Claims, 3 Drawing Figures

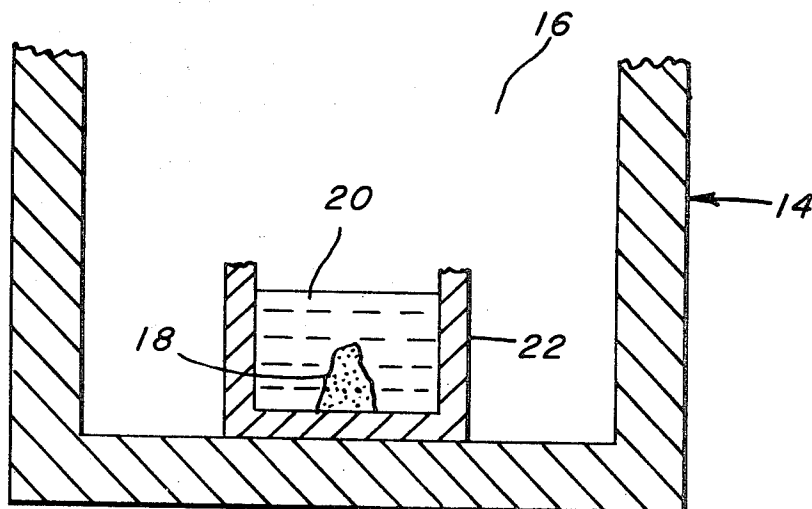


FIG. 1.

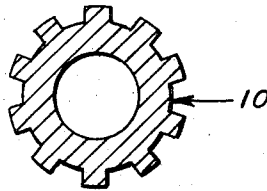


FIG. 2.

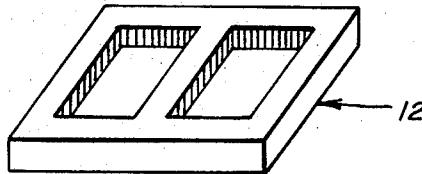
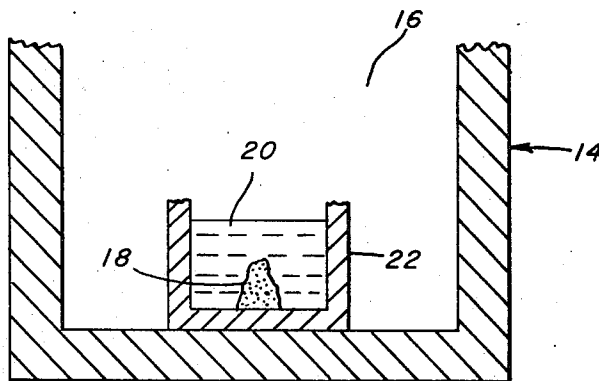


FIG. 3.



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METHOD OF HOT COMPACTING TITANIUM POWDER

BACKGROUND OF THE INVENTION

This invention relates to hot isostatic compacting of titanium powder. More particularly, it relates to the simultaneous use of high temperatures and pressures to compress titanium powder to a high-density, controlled-interstitial product which does not require subsequent forging to improve its microstructure or properties. The term titanium as used herein includes titanium or titanium-base alloys containing at least 50 percent by weight titanium.

Despite continued efforts on the part of research and development personnel, titanium powder metallurgy parts have not reached broad commercial acceptance. The primary unsolved problem until now seems to have been finding a way to produce titanium powder metallurgy parts with properties substantially equivalent to those of titanium forged parts, specifically, commercially acceptable properties, that is, those acceptable for complex parts under most commercial specifications. It has been difficult to obtain parts of the high density, low porosity and high elongation and reduction in area required by wrought titanium specifications. One way in which this has been attempted, but without complete success, has been simultaneous compacting and sintering of titanium powder compacts. For example, a cold compact of loosely pressed titanium powder contained within a suitable mold such as formed by a high temperature-withstanding material has been isostatically compacted at high temperature to produce a product of a simple shape, such as a cylinder or rectangular block. However, such a product was not produced using the necessary controls on interstitials and microstructure. Therefore, it required subsequent forging to improve property levels. Even by such a hot isostatic compact-forge approach it has still been difficult to obtain typical wrought titanium properties.

SUMMARY OF THE INVENTION

After extended investigation we have found that controlling interstitial content, that is, levels of oxygen, nitrogen, hydrogen and carbon, especially oxygen, in the titanium; maintaining for the titanium an alpha-beta microstructure substantially free of alpha-outlined prior beta grains and Widmanstätten patterned alpha particles; keeping the temperature below the beta transus temperature of the alloy undergoing the hot isostatic compacting; and using an intermediate pressure transfer medium between the compacting gas and the powder being compacted and shaped permit attainment of the desired high density, low porosity, high yield and tensile strength, high elongation and reduction of the area, as well as an acceptable controlled limit for final interstitial content, such that the resulting shaped products are commercially acceptable.

According to our invention, to produce a shaped article or product of the desired controlled interstitial content it is desirable to maintain the titanium at a controlled interstitial content throughout the process. The powder which we compact may be prepared by conventional methods, for example, by the process of U. S. Pat. No. 2,628,786, or by machining scrap forgings into chips, using cold water as lubricant and as coolant to inhibit interstitial formation, and then cleaning, drying

and reducing the resultant chips on alcohol by attrition to powder, for example, according to U. S. Pat. No. 3,496,036, using a helium atmosphere. The powder may then be screened to the desired size, for example -40, +200 mesh. Regardless of the method used, however, the heating and working cycle must be such that, as mentioned above, the powder continues to have a microstructure substantially free of alpha-outlined prior beta grains and Widmanstätten patterned alpha particles. We prefer to start with a so-called cold compact, preferably of a density of at least about 60 percent of theoretical, which may be prepared by compacting by conventional methods, one of these being pouring titanium powder stock of about 15-30 percent of theoretical density into a rubber or plastic-type mold having a shape similar to that desired for the final part, drawing a vacuum on the powdered-filled mold, sealing the mold, placing the mold in a cold isostatic compaction unit and compacting same to the starting density. When this procedure is used for forming the compacted powder compressed according to the present invention, the mold may be stripped from the compact after removal of the compact from the cold isostatic compaction unit. If loose, non-compacted powder is used at the start of the hot isostatic compacting, the powder is first poured into a container which may resemble the desired end-product shape, allowing for shrinkage. In this instance, the container may serve as the intermediate pressure transfer medium, provided it is a material adapted for compaction at the required high temperature and pressure used according to the invention.

According to the present invention, the powder or powder compact is placed in a hot isostatic compacting unit. It may be in a container. An intermediate pressure transfer medium is placed around the powder in the desired surface contour, either before or after it is placed inside the hot isostatic compacting unit. The intermediate pressure transfer medium used to form a seal or protective layer between the compact and the gaseous or liquid fluid under pressure used in the hot isostatic compacting unit may be either solid or liquid. Such media tend to promote a uniform pressure distribution against the whole surface of the compact while it is being hot-compacted under pressure. Examples of media which may be used in the compacting operation according to our invention include glass, steel, sand and the like. When in particulate form, they may be either rounded, angular or fibrous. The medium may in the form of a sheath which is sealed over the surface of the compact, for example, steel, preferably stainless. Molten glass is representative of suitable liquid media. Particles of sand may be used as the intermediate pressure transfer medium according to our invention.

According to the preferred practice, a vacuum is drawn on the system containing the cold compact surrounded by the intermediate pressure transfer medium after heating to about 800°-1000° F, and the system is then sealed prior to isostatically compacting to a high density, advantageously greater than about 97 percent.

One of the advantages of this hot isostatic compacting procedure is that the compact may be of almost any desired configuration, including such highly complex forms as gears, marine parts, disc ornamental items or

the like. The cold compact is compressed substantially uniformly in all directions inwardly such that it holds the desired shape. We prefer to use pressures of about 1,000–25,000 psi and temperatures of about 1650°–1850° F. The temperature must be below the beta transus temperature for the particular alloy being compacted such as will not permit formation of the alpha-outlined prior beta grains and Widmanstätten patterned alpha particles which our invention does not allow in the titanium and which prevent attainment according to the invention of the improved ductility comparable to that of forged ingot products. The temperature used is preferably not more than about 200° F below the beta transus temperature for the powder being compacted. The product of the controlled hot isostatic compacting procedure employing intermediate pressure transfer media according to our invention meets final end-use properties that is, commercially acceptable wrought properties when conventionally heat treated. Subsequent forging is unnecessary.

Titanium which may be compacted according to our process includes, for example, in addition to substantially pure titanium, titanium-base alloys, for example, of the following compositions, the numbers referring to percent by weight of the major components other than titanium (except for minor impurities): 6Al–4V, 6Al–6V–2Sn, 8Al–1Mo–1V, 5Al–2.5Sn, 6Al–2Sn–4Zr–6Mo, 6Al–2Sn–4Zr–2Mo–0.25Si and 13V–11Cr–3Al.

According to our invention the titanium powder is preferably hot isostatically compacted at a sufficient temperature and pressure and for a sufficient time to give the resulting shaped articles or products, after heat treatment, typical forged properties, particularly an elongation and reduction in area heretofore generally unattainable for a titanium powder product, for example, for a 1 inch maximum-thickness forged specimen after solution heat treatment and annealing, or annealing a minimum elongation of 10 percent and a minimum reduction in area of 25 percent (after the breaking point when a tensile bar is tested according to the standard procedure), or, after solution heat treatment and aging, a minimum elongation of 8 percent and a minimum reduction in area of 20 percent.

As mentioned above, the compacting temperature according to our invention should not exceed the beta transus temperature. As known to those skilled in the art, this is the temperature above which the titanium or titanium alloy exists chiefly in the beta form. If higher temperatures are used, undesirable alpha-outlined prior beta grains and Widmanstätten patterned alpha, overheated structure, such as may cause unsatisfactory end properties, are likely to develop. As already indicated, the temperature employed is preferably within the range of from 200° F below to the beta transus temperature of the titanium alloy being compacted. When higher temperatures are used, an undesirable microstructure results. Such a microstructure is typical of a crystallographic pattern which indicates low ductility when compared to the microstructure of a forged ingot product. This undesirable microstructure or crystallographic pattern is sometimes characterized by an excessive beta grain size and alpha-outlined prior beta grains, with straight alpha particles within these grains,

or by what we have referred to hereinabove as alpha-outlined prior beta grains and Widmanstätten patterned alpha particles. Presence of such a pattern generally indicates decreased ductility in the product. The high-temperature form of unalloyed titanium, that is, the beta form, has a characteristically body-centered cubic structure and exists above about 1620° F, the beta transus temperature, whereas the low-temperature or alpha form has a characteristically hexagonal-packed structure to which beta transforms below this temperature. The desirable wrought properties brought about by our invention are further associated with a microstructure which comprises equiaxed primary alpha particles within a transformed beta matrix. Use of conventional sintering temperatures after cold compacting tends to destroy the desired microstructure and thereby prevent obtaining the high ductility required for commercial products.

Use of hot isostatic compacting according to our invention substantially prevents any undesirable increase in interstitial content between the starting powder stage and the final shaped product. The interstitial contents for a product of the desired properties are therefore generally substantially the same for the final shaped article produced by hot isostatic compacting according to the invention as for the starting powder or preferred cold compact. For optimum results in producing the desired improved end product properties according to the invention, we prefer that the interstitial content be controlled throughout the process such that at least one of the group oxygen, nitrogen, hydrogen and carbon not exceed a particular value, viz., for a Ti–6Al–4V alloy for oxygen 2,000 ppm or 0.2 percent, for nitrogen 500 ppm or 0.05 percent, for hydrogen 125 ppm or 0.0125 percent or for carbon 1,000 ppm or 0.1 percent, the percent figures referring to percent by weight. That is, we prefer that the content of these interstitials not exceed the requirement of MIL–T–9047.

To decrease porosity problems and to prevent dangerously high interstitial content in the shaped article produced by hot isostatic compacting according to the present invention, it may be advisable to degas by heating prior to the hot isostatic compacting step.

BRIEF DESCRIPTION OF THE DRAWING

For a better understanding of our invention, reference will now be made to the drawing, which forms a part hereof.

FIGS. 1 and 2 are schematic representations of titanium powder metallurgy parts 10 and 12 such as may be produced according to the process of this invention.

FIG. 3 is a schematic cut-away cross-sectional drawing of a portion of a representative hot isostatic compaction unit which may be used according to our invention and which contains therein a titanium powder cold compact inside a surrounding pressure transfer medium which separates it from hot pressurized gas inside the hot isostatic compaction unit. In FIG. 3, a hot isostatic compaction unit is represented by 14, a space containing hot pressurized gas by 16, a powder metal-lurgy shape undergoing or to undergo hot isostatic compacting according to the invention, by 18, an intermediate pressure transfer medium by 20, and a container (for example, stainless steel) by 22. According to

the invention, a powder of controlled <0.2 percent oxygen content 18 is placed in vessel 22 and surrounded by intermediate pressure transfer medium 20. The cold compact pressure medium system is then degassed under vacuum and sealed. It is then placed in hot isostatic compaction unit 14, the unit sealed and the gas 16 pressurized to the desired pressure, for example, 1,000 to 25,000 psi, by means of standard pressurizing methods. The gas may be any suitable gas which is substantially inert to the intermediate pressure transfer medium 20 and the walls of the hot isostatic compaction unit 14, for example, argon. The heating to the desired hot compacting temperature may be done before, during or after gas introduction. The combination of pressure and temperature applied forms the powder to the desired high density shaped article without the necessity of subsequent sintering.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The following example is illustrative of our invention.

Chips were machined from a Ti-6Al-4V alloy billet, using cold water as lubricant and coolant to inhibit interstitial pickup, cleaned with alcohol and reduced to powder by attrition under a cooled argon atmosphere. The resultant powders had an oxygen content of <2,000 ppm. They were screened, while still under the argon atmosphere, to -40, +200 mesh and then placed in a cylindrical rubber bag mold for cold compaction. A vacuum was drawn on the bag and the bag sealed. The powder-filled bag was placed in a cold isostatic compaction unit and compacted at 60,000 psi to a density of approximately 68 percent of theoretical. The cold-compacted sample was removed from the cold isostatic compaction unit and the rubber mold stripped therefrom. It was then sheathed in a stainless steel container which served as an intermediate pressure transfer medium, a vacuum was drawn on the compact-container system, and the container sealed. The stainless steel-clad cold compact was placed in a hot isostatic compaction unit such as that depicted in FIG. 3 for six hours at 1600° F and 1,500 psi and isostatically compacted therein. The resultant hot isostatically compacted sample was then quartered, one quarter being retained as hot isostatically compacted, and the remaining three upset either 10, 20 or 30 percent on flat dies at 1750° F. Tensile blanks were machined from each quarter and solution heat treated and aged as follows: heat one hour at 1750° F, water quench within five seconds, age four hours at 1000° F and air cool. Tensile data were taken and compared to minimum forging commercial requirements for similar prior art standard forgings. These appear in the following table.

TABLE

[Tensile properties of solution heat treated and aged samples of hot isostatically compacted Ti-6Al-4V alloy powders]

Sample	Percent upset	Yield strength, K.s.i.	Tensile strength, K.s.i.	Ductility, percent	
				Elongation	Reduction in area
1.....	0	157.5	176.0	12.5	37.7
2.....	10	161.8	177.9	14.1	36.8
3.....	20	161.2	178.4	12.5	37.7
4.....	30	161.5	177.9	12.5	38.2
Aerospace material ¹		160.0	160.0	8.0	20.0

¹ Specification (AMS) 4,967 minimums.

² Plus.

Metallographic examination showed the hot isostatically compacted and solution heat treated and aged samples to be of the desired equiaxial primary alpha particles within a transformed beta matrix structure. As indicated in the above table, the properties of the hot isostatically compacted samples after solution treatment and aging were well above commercial specifications.

While the invention has been described in terms of preferred embodiments, the claims appended hereto are intended to encompass all embodiments which fall within the spirit of the invention.

Having thus described our invention and certain embodiments thereof, we claim:

1. A process for production of a shaped article having properties comparable to those of conventionally forged titanium wrought articles without the need for subsequent sintering operations or mechanical working operations to substantially strengthen the article, said process comprising hot isostatically compacting titanium powder at a temperature in the region of but below the beta transus temperature of the titanium, employing in said compacting step a fluid which is separated from said powder by means of an intermediate pressure transfer medium through which a substantially uniform pressure is transmitted substantially evenly peripherally, and maintaining in said titanium throughout the process a microstructure which is substantially free of alpha-outlined prior beta grains and Widmanstätten patterned alpha particles, thereby forming a shaped article of high density and controlled interstitial content and having an elongation, reduction in area and strength levels comparable to the elongation, reduction in area and strength levels of similar forged titanium ingot.

2. The process of claim 1 wherein the interstitial content is controlled by maintaining the oxygen content of said titanium throughout the process at not exceeding the requirement of MIL-T-9047 on the filing date of this specification.

3. The process of claim 1 wherein the interstitial content is controlled by maintaining the nitrogen content of said titanium throughout the process at not exceeding the requirement of MIL-T-9047 on the filing date of this specification.

4. The process of claim 1 wherein the interstitial content is controlled by maintaining the hydrogen content of said titanium throughout the process at not exceeding the requirement of MIL-T-9047 on the filing date of this specification.

5. The process of claim 1 wherein the interstitial content is controlled by maintaining the carbon content of said titanium throughout the process at not exceeding the requirement of MIL-T-9047 on the filing date of this specification.

6. The process of claim 1 wherein the temperature at which the hot compacting takes place is between about 200° F below the beta transus temperature and the beta transus temperature of the titanium.

7. The process of claim 1 wherein the compacting pressure is from about 1,000 to about 25,000 psi.

8. The process of claim 1 wherein the density of the shaped article exceeds about 97 percent of theoretical.

9. The process of claim 1 wherein the powder is compacted to at least about 60 percent theoretical density by cold compacting before said hot compacting.

10. The process of claim 9 wherein the cold compacting is followed by degassing prior to said hot compacting.

11. The process of claim 1 wherein the intermediate pressure transfer medium is selected from the group consisting of molten glass, sand and steel.

12. The process of claim 1 wherein the intermediate pressure transfer medium is solid.

13. The process of claim 1 wherein the intermediate pressure transfer medium is liquid.

14. The process of claim 1 wherein, prior to the hot compacting, the titanium powder is prepared by filling

a flexible mold of substantially the shape of the resulting shaped article with titanium powder of a density of about 15-30 percent of theoretical and cold isostatically compacting said powder in such mold to a density of at least about 60 percent of theoretical.

15. The process of claim 14 wherein, prior to the hot compacting, the cold-compacted powder is degassed by heating.

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