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METHOD OF HEAT-TREATING BETA TITANIUM-BASE ALLOYS

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ABSTRACT OF THE DISCLOSURE

Beta titanium-base alloys containing 15–35% molybdenum, 15–35% vanadium, up to 5% zirconium, .01–0.2% boron, 0.1–5% hafnium, .01–.2% carbon, up to 5% of at least one element selected from the group consisting of W, Ta, Nb, Cr and Al and the balance Ti are strengthened by heating the alloy to a temperature between 1300° C. and 1500° C. at which the precipitate-forming elements dissolve in a beta matrix, cooling the alloy to retain the precipitate-forming elements in solution in the matrix and subsequently ageing the alloy at a temperature between 500° C. and 950° C. to form a very fine precipitate.

This invention relates to a method of heat-treating beta titanium-base alloys.

In prior patent application Ser. No. 463,573, filed June 14, 1965, there is described beta titanium-base alloys having good creep strength containing, by weight, 15–35% molybdenum, 15–35% vanadium, the total molybdenum plus vanadium content being in the range 40–55%, up to 8% zirconium, 0.01–0.2% boron, balance titanium. Incidental alloying elements which may also be present include 0–5% tungsten, tantalum, niobium, chromium and aluminum. In such alloys 0.1–5% hafnium and 0.01–0.2% carbon may also be present. In such alloys, carbon and boron combine with the other elements present, notably zirconium to form complex precipitates. Such alloys are hereinafter referred to as "alloys of the kind described."

In our prior patent application we have described a treatment for alloys of the kind described in which the alloys are hot-worked, at temperatures between 1000° C. and 1200° C. and subsequently reheated for a period at 900° C. and air-cooled. Such treatment was found to be unreliable in that the best creep properties of the alloy were not obtainable consistently or reproducibly and for applications such as aircraft engines, the alloys used must be consistent in their creep properties.

We have discovered that alloys of the kind described are strongest when the precipitates are finely and uniformly distributed in the beta matrix and that the required particle size and distribution can be consistently produced by the treatment now to be described.

According to the invention there is provided a method of treating a beta titanium-base alloy of the kind described in which the alloy is heated at a temperature at which the precipitate-forming elements are dissolved in the beta matrix of the alloy to nucleate the matrix with the maximum number of nucleation sites for precipitation, and the alloy cooled at a rate sufficient to retain the precipitate-forming elements in solution and forming a fine precipitate at the nucleation sites.

The solubility of carbon and boron in these alloys increases with increasing temperature, so that most of the carbon and boron is in solution at temperatures above about 1100° C., which thus defines a lower limit for the solution treatment temperature. Solution of carbon and boron is complete in laboratory samples after only short times at temperatures above 1100° C., but solution treatment times will in practice be suited to section size, according to accepted practice in titanium metallurgy. Solution treatment may be terminated either by cooling in air or by quenching in one of the several liquids which are in general use.

The matrix may be nucleated with the maximum number of sites by quenching or by working the material by forging or extrusion following solution treatment.

Precipitation is carried out by heating at temperatures between 500 and 950° C., the time required to produce a suitable precipitate decreasing with increasing temperature. Alternatively, the precipitate can be formed during air cooling after hot working at the solution treatment temperature or during warm working after solution treatment and quenching. The product is strongest when the precipitates are so finely divided that they are only resolvable in the electron microscope according to well-established principles. It is necessary, therefore, to provide as many nucleation sites as possible for precipitation. On the other hand, if precipitation has been carried out at too high a temperature or for too long a time at a certain temperature, the particles grow too large and do little to strengthen the alloy. Examples of the effect of heat treatment, as described above, on the strength of the alloys as measured by stress-rupture tests are shown in Table I.

TABLE I

Alloy composition (Wt. percent)	Heat treatments A 10 mins. at 1,300° C., WQ 7 hrs. at 650° C.	Heat treatments B Warm-rolled at 800° C., 24 hrs. at 625° C.
Ti-20Mo-20V-4Zr-2Hf-0.03C	0.0004	0.036
Ti-20Mo-20V-2Hf-0.03C	0.0069	0.100
Ti-20Mo-20V-2Hf-0.03C-0.03B	0.012	0.084

Values of total strain (inch per inch) in 5 hours at a stress of 20 tons/sq. in. at 575° C. are given in the table.

It will be seen from the table that the treatment B produces inferior results to treatment A. These results may be explained in terms of the theory of dispersion hardening as briefly outlined above. In treatment A, precipitates are dissolved at 1300° C. and precipitated uniformly and in a finely divided form on subsequent ageing at 650° C. In treatment B, although a high density of nuclei is created by warm-working at 800° C., the carbide formers are not in solution prior to warm-working and are not mobile enough to redistribute on the nucleation sites during ageing. Treatment A is typical of the heat-treatments that strengthen the alloy. Further more detailed examples of the effect of heat treatment are given in Table II. The alloy containing Ti-20Mo-20V-4Zr-0.05B, which contained .03 carbon as impurity, was tested in the warm rolled and annealed condition and after solution treatment at 1400° C., water quenching and ageing for 1 hour at 650° C. The strain after 100 hours in a stress-rupture test in the warm rolled and annealed condition was 13.2%, while after solution treatment and ageing, the strain after 100 hours was less than 0.1%. An alloy containing Ti-22Mo-22V-4W-4Zr-0.03C (total carbon including impurity 0.06%) was tested after warm rolling at 1000° C. and after extrusion at 1370° C. In the as-rolled condition the strain at 100 hours was 6%, while after extruding at 1370° C. the strain after 100 hours was less than 0.1% and after 250 hours was a 0.2%. These results may be explained in terms of the theory of dispersion hardening. During warm rolling, although a large number of nucleation sites are created, the precipitate-forming elements are not sufficiently mobile to redistribute. Solution treatment, on the other hand, dissolves all of the precipitates in the electron micrograph, while subsequent ageing at 650° C. causes the precipitation of a fine dispersion of particles resolvable at a magnification of 30,000 diameters. Similarly, during extrusion at 1370° C., all the precipitate-forming elements are dissolved during the soak prior to extrusion, during extrusion a large number of nucleation sites are produced and the precipitates form on these sites during cooling following extrusion.

TABLE II

Alloy composition	Condition	Strain in stress-rupture test at 20 t.s.i., 575° C. 100 hrs., percent	Strain in stress-rupture test at 20 t.s.i., 575° C. 250 hrs., percent
Ti-20Mo-20V-4Zr-0.05B	Warm rolled and annealed.	13.2	
	20 mins. at 1,400° C., water quench, 1 hr. at 650° C.	<.1	
Ti-22Mo-22V-4W-4Zr-0.03C.	Warm rolled	6	
	20 mins. at 1,370° C., extruded at 1,370° C., air cooled.	<.1	.2

Treatments in accordance with the present invention must be carried out within the limits now to be specified.

Solution treatment and working is carried out at a temperature above 1100° C., but not greatly in excess of 1450° C. (1500° C. as a maximum) to avoid undue oxidation. If the material is then worked from the solution treatment temperature, sufficient nucleation sites are created during working and a fine precipitate forms during air cooling following working. If the material is not worked after solution treatment, the cooling rate must be fast enough to retain the precipitate-forming elements in solution down to the ageing temperature. With laboratory samples, air cooling is sufficient, but with larger samples quenching in one of the several liquids which are in general use is necessary. Ageing is then carried out at temperatures above 500° C., below which the diffusion of the precipitate-forming elements is so slow as to preclude their formation. The upper limit for ageing is that temperature at which undue coarsing of the precipitate occurs, which for these alloys is approximately 950° C. Ageing time is selected in accordance with temperature, the higher temperatures requiring shorter times for the formation of the fine precipitate. Within the temperature range specified, these times may be measured in terms of hours.

We claim:

1. A method of heat-treating a beta titanium-base alloy consisting essentially of, by weight, beta matrix-forming elements molybdenum 15–35% and vanadium 15–35%, the total of said molybdenum and vanadium together being 40–55%, up to 5% zirconium, 0.01–0.2% boron, up to 5% of at least one metal selected from the group consisting of tungsten, tantalum, niobium, chromium and aluminum, and precipitate-forming elements hafnium 0.1–5% and carbon 0.01–0.2%, balance titanium, said method comprising the steps of

(a) heating said alloy at a temperature between 1300° C. and 1500° C. to effect solution of said precipitate-forming elements in said beta matrix whereby said beta matrix is nucleated with a maximum number of nucleation sites,

(b) cooling said alloy at a rate sufficient to retain said precipitate-forming elements in solution in said beta matrix,

(c) ageing said alloy for a sufficient time at a temperature between 500° C. and 950° C. to form a fine precipitate at said nucleation sites.

2. A method of heat-treating a beta titanium-base alloy consisting essentially of, by weight, beta matrix-forming elements molybdenum 15–35% and vanadium 15–35%, the total of said molybdenum and vanadium together being 40–55%, up to 5% zirconium, 0.01–0.2% boron, up to 5% of at least one metal selected from the group consisting of tungsten, tantalum, niobium, chromium and aluminum, and precipitate-forming elements hafnium 0.1–5% and carbon 0.01–0.2%, balance titanium, said method comprising the steps of

(a) hot working said alloy at a temperature between 1300° C. and 1500° C. to effect solution of said precipitate-forming elements and to form a maximum number of nucleation sites in said beta matrix,

(b) cooling said alloy at a rate sufficient to retain said precipitate-forming elements in solution in said beta matrix,

(c) precipitating a fine precipitate on said nucleation sites at a temperature between 500° C. and 950° C.

3. A method of heat-treating a beta titanium-base alloy consisting essentially of, by weight, beta matrix-forming elements molybdenum 15–35% and vanadium 15–35%, the total of said molybdenum and vanadium together being 40–55%, up to 5% zirconium, 0.01–0.2% boron, up to 5% of at least one metal selected from the group consisting of tungsten, tantalum, niobium, chromium and aluminum, and precipitate-forming elements hafnium 0.1–5% and carbon 0.01–0.2%, balance titanium, said method comprising the steps of

(a) heating said alloy at a temperature of 1300° C.,

(b) quenching said alloy in water,

(c) ageing said alloy at 650° C.

4. A method of heat-treating a beta titanium-base alloy consisting essentially of, by weight, beta matrix-forming elements molybdenum 15–35% and vanadium 15–35%, the total of said molybdenum and vanadium together being 40–55%, up to 5% zirconium, 0.01–0.2% boron, up to 5% of at least one metal selected from the group consisting of tungsten, tantalum, niobium, chromium and aluminum, and precipitate-forming elements hafnium 0.1–5% and carbon 0.01–0.2%, balance titanium, said method comprising the steps of

(a) heating said alloy at a temperature between 1300° C. and 1500° C. at which said precipitate-forming elements dissolve in said beta matrix,

(b) cooling said alloy at a rate sufficient to retain said precipitate-forming elements in solution in said beta matrix,

(c) warm-working said alloy,

(d) ageing said alloy at a temperature between 500° C. and 950° C. to form a fine precipitate at said nucleation sites.

5. A method of treating an alloy consisting of, by weight 20% molybdenum, 20% vanadium, 4% zirconium, 0.05% boron, balance titanium and impurities comprising heating the alloy at 1400° C., water quenching and ageing for 1 hour at 650° C.

6. A method of treating an alloy consisting of, by weight 22% molybdenum, 22% vanadium, 4% tungsten, 4% zirconium, 0.3% carbon, balance titanium and impurities, comprising heating and extruding the alloy at 1370° C. and air cooling at a rate suitable for the formation of a fine precipitate.

7. A method of treating an alloy selected from the group consisting of an alloy consisting of, by weight 20% molybdenum, 20% vanadium, 4% zirconium, 2% hafnium, 0.3% carbon, balance titanium, an alloy consisting of 20% molybdenum, 20% vanadium, 2% hafnium, 0.03% carbon, balance titanium, an alloy consisting of 20% molybdenum, 20% vanadium, 2% hafnium, 0.03% carbon, 0.03% boron, balance titanium, which comprises heating the alloy at 1300° C., water-quenching and ageing at 650° C. for 7 hours.

8. A method as in claim **1** wherein the precipitate formed during said ageing step is resolvable only with an electron microscope.

9. A method as in claim **1** wherein the precipitate formed during said ageing step is resolvable only at a magnification of at least 30,000 diameters.

10. A method as in claim **2** wherein said hot working step includes an extrusion step.

11. A method as in claim 2 wherein said hot working step includes a forging step.

12. A method as in claim **4** wherein said warm working is carried out at about 1000° C.

References Cited

UNITED STATES PATENTS

2,754,203	7/1956	Vordahl ------------	75—175.5
2,754,204	7/1956	Jaffee et al. --------	75—175.5
3,306,739	2/1967	Evans et al. --------	75—175.5

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148—32.5, 133