United States Patent Office

2,950,192

Patented Aug. 23, 1960

2,950,192

PRODUCTION OF WROUGHT TITANIUM BASE ALLOYS AND RESULTING PRODUCT

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No Drawing. Filed Apr. 21, 1954, Ser. No. 424,569

10 Claims. (Cl. 75—175.5)

This invention pertains to strong and ductile wrought titanium base alloys, characterized by a microstructure consisting of a coherent admixture of small bodies of alpha titanium and beta titanium, and to methods of producing the same, and to wrought articles thereof.

The invention pertains more particularly to wrought titanium base alloys of the character aforesaid, containing about 2.5 to 15 atomic percent in total, of one or more elements selected from the group consisting of manganese, chromium, molybdenum and iron. The binary alloys of the invention will thus, of course, contain about 2.5 to 15 atomic percent of either manganese, chromium, molybdenum or iron. The corresponding weight percents are approximately 3–16% each for iron and manganese, about 3–15% for chromium, and about 5–25% for molybdenum. For the ternary and higher order alloys, the total content of these elements shall be, as above stated, about 2.5 to 15 atomic percent, with lower effective limits for the individual elements of about 1% by weight each for molybdenum, manganese and iron and about 3% by weight for chromium. The alloys of the invention are strengthened without undue embrittlement by additions of up to about 0.5% carbon, up to about 0.3% of oxygen, and up to about 0.1% nitrogen.

These alloys may be produced by arc melting in a cold-mold furnace, in an inert atmosphere such as argon, or by equivalent procedures. The titanium employed may be either the high purity "iodide" titanium or the commercial purity product obtained by the magnesium reduction of titanium tetrachloride, or an equally pure material as obtained by other procedures.

This application is a continuation-in-part of my copending applications Serial No. 132,327, filed December 10, 1949, now abandoned, and Serial No. 229,143, filed May 31, 1951, and of the joint application of Walter L. Finlay and myself Serial No. 79,556 filed March 4, 1949, and now abandoned.

The low temperature or alpha phase of substantially pure titanium, which is of close-packed hexagonal structure, transforms at a temperature of about 885° C., to the high temperature or beta phase, which is of body-centered cubic structure. The presence of such alloying elements as carbon, oxygen, nitrogen and aluminum tends to raise the beta transformation temperature, and establish a relatively narrow zone or field of mixed alpha-beta structure. The presence of certain other alloying metals, prominent among which are iron, chromium,

manganese and molybdenum, has a quite different effect upon transformation. Increasing amounts of these alloying metals stabilize the beta phase at progressively lower temperatures, and establish a mixed alpha-beta field of substantial scope.

The present invention comprises, in one of its aspects, the discovery of a method for imparting to titanium base alloys of the type aforesaid an interleaved dispersion or distribution of the alpha and beta phases, and of a unique microstructure resulting therefrom. An essential step in the method is the plastic deformation of the alloy at a temperature within the two-phase or mixed alpha-beta field. Another essential step is the selection of a titanium base alloy composition possessing two or more ductile phases stable over a suitable temperature range. Such plastic deformation of such a structure differs widely from the generally accepted practice of deforming to a desired size and configuration in a single phase field, and subsequently heat treating to secure a desired set of properties. In the practice of applicant's method, the essentials of the desired structure are established by plastic deformation, and while subsequent heat treatment is permissible and may be further beneficial, it must be so controlled as not to destroy the essential structure set up by plastic deformation.

The alloys amenable to the present invention are of the mixed-phase structure. They are characterized by the presence of two phases of unequal strength, both of which possess some ductility but which deform by basically different crystallographic mechanisms. Thus, a stress concentration in one phase tends to diffuse, rather than to propagate, when it encounters the other phase. The titanium base alloys particularly disclosed by this specification possess all of the unusual set of qualifications: their alpha phase is relatively weak but quite ductile; their beta phase is relatively strong while nevertheless maintaining some ductility; and alpha, being a hexagonal close-packed structure, deforms in a basically different manner than does beta, which has a body-centered cubic structure.

The present invention is particularly directed to sheet, wire and rod products in which service stresses are applied substantially parallel to the rolling or drawing direction and in which, therefore, the optimum distribution of the weaker phase is in thin fibrils or platelets whose longitudinal axes are parallel to the rolling or drawing direction. The presence of the weaker phase in thin fibrils or platelets disposed parallel to the working direction, presents a minimum area of weaker phase normal to the applied stress and so constitutes the strongest distribution. Conventional processing procedures such as working in the all-beta region followed by cooling at any convenient rate produce a distribution of the weaker phase which presents an appreciably greater area of weaker phase normal to the applied stress than that achieved by the novel processing disclosed in this invention and so is weaker. A further important advantage of the optimum distribution is that a stress concentration arising in the stronger phase (which is making the major contribution to supporting the applied stress) tends, in starting to propagate across the fibrils and platelets of the weaker phase, to be diffused in a manner analogous to the reaction to stress of a cable in contrast to that of a rod.

Each of the phases in the optimum distribution disclosed by this invention may be either continuous or discontinuous in the worked direction. The stronger phase should predominate in order to give higher strength and thus tends to be continuous in the worked and, therefore, the loadbearing direction. Even if it is not continuous, however, its fibrils and platelets are coherent with their contiguous material and so, like the fibrils in a textile thread, act as if they were continuous. It is an essential feature of this invention that, normal to the direction of working, neither phase is continuous.

The history of the alloy prior to plastic deformation in the two-phase field is of little, if any, significance. The effect of plastic deformation in the two-phase field is to increase ductility at a given strength level, and this effect is quite independent of prior history. Whether the specimen has been hardened by quenching from a beta field or lower temperature, or has been fully annealed by slow cooling from a beta field temperature, or given any other treatment, extensive working in the two-phase field still develops its optimum properties, viz., the highest ductility at a given strength level. For example, the quenching from the all-beta field of a binary alloy of 4% manganese, balance substantially all commercial purity titanium, results in a structure characterized by extended martensite-like plates of alpha and irregular bodies of retained beta. The quenching from the all-beta field of a similar alloy containing about 7% manganese produces unstable beta grains. On subsequent elevated temperature aging or service there is a tendency to the growth of continuous bodies of alpha at the grain boundaries. Both structures are predisposed to fracture along the extended region of relatively weak alpha. If either alloy is slow cooled it becomes "dead soft" and is, for most uses, in its least serviceable condition. Extensive two-phase working of either of these alloys from either the quenched or the annealed condition develops properties identical with those developed by working the same alloy from the other condition.

Extensive plastic deformation such as can be obtained in rolling sheet or rod or in drawing wire at a temperature within the two-phase field has the effect not only of breaking up and dispersing the continuous masses, particularly the relatively weak alpha, and reducing such continuous masses to much smaller, substantially discontinuous and discrete particles, but also of producing the interleaved structure of fibrils and platelets of alpha and beta which is disclosed by this specification as being optimum. If one phase predominates, it tends to form a matrix in which island platelets or fibrils of the second phase are embedded.

For example in a sheet of an alloy of commercial purity titanium with 4.1% manganese after a 90% area reduction by rolling at a temperature of 650° C. and stabilized for one hour at a temperature of 550° C., the dispersion and discontinuity of the phases, particularly the alpha phase, are substantially optimum for sheet, a good balance being struck between properties transverse and parallel to the working direction. The platelets are arranged parallel to the sheet surfaces. In a wire, the fibrils would be arranged parallel to the axis, and the structure is comparable with that of a cable as compared with a solid rod. The individual fibers of the stronger but more brittle phase can, to some extent, yield separately and thus distribute the load throughout the whole, while the effect of stress-raising voids, lattice imperfections, and the like, tends to be nullified at the first junction with the weaker and more ductile phase.

A typical specimen of the 4.1% manganese alloy has a yield strength in excess of 120,000 p.s.i., an ultimate strength above 145,000 p.s.i., an elongation in ½" of 16%, and a bend ductility of 3.3 T.[1] This ratio of ductility-to-strength is definitely superior to any obtainable from this alloy without extensive plastic deformation at a temperature within the two-phase field. The same is true of the numerous other alloys which have been found amenable to the processing of this invention. An alloy of 7% manganese, balance substantially all commercial purity titanium, after 75% area reduction by rolling at a temperature of 650° C., shows a structure substantially similar to that of the 4.1% manganese alloy. Prior to such warm working and after furnace cooling from 750° C., it showed a heterogeneous mass of variously oriented extended bodies of the two phases. After 75% warm rolling and stabilization for one hour at 550° C., alloys of commercial titanium with about 8% manganese show a yield strength of about 145,000 p.s.i., an ultimate strength over 157,000 p.s.i., an elongation in ½" of about 16%, and an average bend ductility of about 3 T.

While the broad invention contemplates plastic deformation at any temperature within the two-phase field at which the strength of the alloy permits such deformaion, the working temperature should not too closely approach the boundary temperature between the mixed alpha-beta field and the beta field. The heating of the alloy prior to working, and possibly local heating induced by working itself promote transformations toward equilibrium at the particular temperature. If the working temperature is too close to the all-beta temperature, the tendency to beta formation is so great that a substantial part of the benefits of the invention may be lost. In general, the working temperature should not approach the all-beta temperature within about 50° to 100°. The preferred temperature range for plastic deformation is from about 400° C. to about 50–100° C. below the beta transus temperature.

The beta transus temperature for any particular alloy in accordance with the invention may be determined by quenching specimens from progressively higher temperatures, until a completely martensitic or retained beta micro-structure is obtained.

Increasing amounts of plastic deformation within the two-phase field effect increasingly complete dispersion and interleaving of the phases, and proportionately increase ductility at a given strength level. Generally stated, strength is a function of composition and conditions of stabilization subsequent to two-phase temperature working. For an alloy of given composition, it has been found possible, by appropriate stabilization, to maintain a substantially constant strength-level throughout varying amounts of plastic deformation. Any material variations in strength being thus eliminated, the progressive increase in ductility with increasing two-phase temperature deformation has been established, without complication by other variables. The experimental work leading to the present invention was exhaustive, comprising different alloys, different treatments prior to two-phase working, different working temperatures, and varying mounts of two-phase temperature deformation.

As an example of the work, the following series of tests is described. Three alloys were selected which contained, respectively, 3.5%, 4.1% and 4.7% manganese, balance substantially all commercial purity titanium. An ingot of each composition was forged at 870° C. to a ½" to ⅝" slab, each slab was cleaned and rolled at about 815° C., to sheet of four different thicknesses so chosen that the varying amounts of warm rolling reduction (to be described) resulted in specimens of the same thickness, i.e., all finished specimens received the same total reduction from the ingot. Each of the twelve sheets was subdivided into three parts, and the three parts respectively of each sheet were conditioned

[1] The measurement of bend ductility is not standardized. The present applicant and his associates measure this property as the radius over which the specimen can be bent to an angle of 75° without cracking, the radius being expressed as a multiple of specimen thickness.

for two-phase temperature rolling by three different treatments, as follows:

Condition "Q" Heated in air one hour at 815° C., water quenched;

Condition "Q–T" Heated in air one hour at 815° C., water quenched, and tempered sixteen hours at 550° C.;

Condition "F–C" Heated in air one hour at 815° C., furnace cooled.

All specimens were then rolled to a uniform thickness at a two-phase field temperature, three different temperatures being used. The thickest specimens of each set were reduced 90%, the next thinner 75%, the next thinner 60%, and the thinnest 30%, the final rolled thickness of all being the same. All specimens were then stabilized for one hour at 550° C. Strength and ductility were then measured, and found to be as follows:

Composition: 3.5% manganese—balance, commercial titanium.
Rolling temperature: 650° C.
Stabilized: 1 hour at 550° C., after rolling.

Pre-Rolling Condition	Reduction, percent	Yield (1,000s p.s.i.)	Ultimate (1,000s p.s.i.)	Elongation, percent	Bend Ductility (Least Favorable Direction)
Q	30	107	136	15	7.7
Q	60	106	134	19	5.1
Q	75	102	130	20	3.1
Q	90	103	130	21	2.6
Q–T	30	102	131	17	9.0
Q–T	60	100	131	19	4.3
Q–T	75	96	126	21	2.5
Q–T	90	99	125	23	2.6
F–C	30	102	131	16	6.3
F–C	60	100	130	19	3.7
F–C	75	98	126	21	3.1
F–C	90	100	124	23	3.1

Composition: 4.7% manganese—balance, commercial titanium.
Rolling temperature: 700° C.
Stabilized: 1 hour at 550° C., after rolling.

Pre-Rolling Condition	Reduction, percent	Yield (1,000s p.s.i.)	Ultimate (1,000s p.s.i.)	Elongation, percent	Bend Ductility (Least Favorable Direction)
Q	30	119	137	8	10
Q	60	128	141	6	10
Q	75	121	136	17	6.4
Q	90	112	132	19	3.1
Q–T	30	124	138	----------	10
Q–T	60	124	137	6	6.2
Q–T	75	121	136	9	3.4
Q–T	90	111	132	21	2.3
F–C	30	110	134	6	6.6
F–C	60	115	138	13	6.6
F–C	75	111	135	16	3.1
F–C	90	113	135	17	2.3

Composition: 4.1% manganese—balance, commercial titanium.
Rolling temperature: 565° C.
Stabilized: 1 hour at 550° C., after rolling.

Pre-Rolling Condition	Reduction, percent	Yield (1,000s p.s.i.)	Ultimate (1,000s p.s.i.)	Elongation, percent	Bend Ductility (Least Favorable Direction)
Q	30	117	140	3	10
Q	60	130	144	3	10
Q	75	128	141	7	7.6
Q	90	125	139	12	6.2
Q–T	30	110	136	4	10
Q–T	60	114	135	12	7.8
Q–T	75	119	134	14	6.4
Q–T	90	123	134	18	3.8
F–C	30	107	132	6	10
F–C	60	111	134	12	6.4
F–C	75	109	132	14	4.6
F–C	90	113	132	18	3.9

In the foregoing tables, each of the reported values is the average of four different tests. The temperature of 815° C., to which all of the alloys were heated prior to the two-phase rolling, if not within the all-beta field lies so close to the all-beta field as to destroy the effect of previous working. The tabulated results definitely show for each alloy rolled from each of the three pre-rolling conditions, first, a negligible variation in yield and ultimate strength with varying amounts of two-phase temperature rolling; and, second, a progressive increase in ductility for increasing amounts of such rolling. These effects are the same at any of the three rolling temperatures. Other variables being eliminated, increasing amounts of plastic deformation in the two-phase field effect a progressive increase in ductility.

Stabilization, as above described, is desirable under most conditions, and can be performed at temperatures up to about 550° C., without materially altering the microstructure.

While the optimum combination of strength and ductility is secured by extensive plastic deformation in the two-phase field, fabrication problems frequently render it impossible to roll sheet to the strength level desired in the fabricated product. Bending, forming, drilling, and like operations, cannot be satisfactorily performed at the desired high strength. It has been found that the alloys processed according to the present invention can be annealed to the low strength level necessary for fabrication, and, after fabrication, quenched to the desired high strength, and still retain the essentials of the dispersed phase structure. The alpha phase is spheroidized, but remains as discrete bodies rather than continuous masses.

An alloy containing 6.8% manganese, balance commercial purity titanium, which, after extensive plastic deformation in the two-phase field, had been heated for 1 hour at 700° C., and water quenched, was found to consist of a beta matrix containing very numerous relatively small and substantially discrete globules of alpha. This specimen, after stabilization for 1 hour at 500° C., showed a yield strength of about 140,000 p.s.i., an ultimate strength of about 170,000 p.s.i., an elongation in ½" of about 12%, and a bend ductility of about 3 T.

While in the foregoing description the binary alloys of titanium and manganese have been used for the purpose of illustration, it has been found as the result of very extensive work that the novel processing is equally applicable to any alloys comprising two ductile phases, both of which are relatively stable at normal temperatures. Prominent among these are the binary alloys of titanium with molybdenum, chromium and iron, as well as manganese, and the ternary and higher alloys of titanium with the metals of this group, and with aluminum up to about 8%. While in the binary titanium-aluminum alloys the beta phase is not stable at normal temperatures, it is stabilized by the addition of one or more of the metals, manganese, molybdenum, chromium and iron. The weight percentages of the different metals of the group, manganese, molybdenum, chromium and iron, which, when added to titanium produce a two-phase structure, vary with the particular metal—the maximum for manganese, for example, being about 16%, but the atomic percentages fall within the range of about 2.5 to 15 atomic percent. Below about 2.5 atomic percent the beta phase is either absent altogether or present only in a negligible amount; while above about 15 atomic percent the alloys show an all-beta structure on quenching. Typical alloys and their properties as worked in the two-phase field, both with and without subsequent stabilization, are as follows: minimum tensile strength of at least 137,000 p.s.i. and minimum elongation of at least 2%.

Composition Percent (Balance Titanium)	Tensile Properties							
	As Warm Rolled in the Alpha-Beta Field				As Stabilized at 600° C.			
	.2% Yield	Ultimate	Elongation in ½″	Bend Radius	.2% Yield	Ultimate	Elongation in ½″	Bend Radius
3.4Mn—0.23C	153,000	173,000	12	6.5	138,000	149,000	18	3.3
4.4Mn—0.22C—0.1N	152,000	175,000	13	6.5	144,000	160,000	22	4.9
6.1Mn—0.12C—0.03N	159,000	184,000	2	4.1	132,000	146,000	12	2.9
13.2Mn—0.15C	170,000	187,000	6	6.2	155,000	160,000	12	4.0
7.4Cr—0.34C*	134,000	154,000	7	4.6	132,000	143,000	14	2.7
12.4Cr—0.17*	141,000	160,000	5	7.0	133,000	142,000	11	2.7
2.4Fe—0.32C—0.02N	117,000	146,000	12	2.7	98,000	125,000	19	1.7
5Fe—0.25C	138,000	164,000	8	8.8	119,000	141,000	8	6.5
4.7Mo—0.2C—0.1N	135,000	159,000	16	7.5	122,000	145,000	20	6.2
3.4Mn—2.8Al—0.51C—0.01N	140,000	170,000	8	6.1	135,000	159,000	10	5.8
3.1Mn—4.1Al—0.11C—0.03N	127,000	154,000	14	4.8	117,000	134,000	21	2.0
3.4Mn—0.8Mo—0.32C—0.03N	136,000	164,000	8	4.9	127,000	139,000	19	2.7
2.5Mn—1.6Fe—0.2C	155,000	175,000	10	6.5	138,000	150,000	20	6.6
5.5Mn—1Al—6.1Cr—0.32C	155,000	178,000	4	7.5	149,000	160,000	14	4.4
6Mn—4.3Cr—2.9Mo—0.42C	153,000	171,000	6	7.3	148,000	158,000	13	6.2
3.3Mn—1Fe—4Al—0.2C—0.02N	127,000	158,000	10	5.7	118,000	138,000	15	2.2
8.2Mn—1.0Fe	163,000	169,500	11.7	------	------	------	------	------
9.4Mn—5.2Mo	128,100	146,100	7	------	------	------	------	------
9.1Mn—5Cr—5.1Mo	135,700	158,400	10	------	------	------	------	------
5.1Mo—10.1Mn—5.2Cr	138,900	144,600	23	------	------	------	------	------
9.3Mn—5.1Cr—4.7Mo	197,000	215,800	2	------	------	------	------	------
4.7Mn—5Cr—4.7Mo	158,700	183,800	4	------	------	------	------	------
90Mn—5Cr**	184,800	197,900	3	------	------	------	------	------

*Stabilized at 700° C.
**Cold worked.

In my copending application Serial No. 132,328, filed December 10, 1949, now U.S. Patent No. 2,704,251, dated March 15, 1955, I have shown that binary alloys of titanium and manganese containing about 6 to 12% manganese are characterized by excellent strength and ductility. In the present application I have shown that the binary titanium-manganese alloys containing from about 3% to less than 6% manganese, when produced in wrought form by plastic deformation in the alpha-beta field likewise possess high strength and ductility.

The alloys of the invention having tensile elongations as low as about 2% are useful in massive form, for example, forgings, and are useful in the form of rolled sheet or drawn wire with minimum bend ductilities as high as 20 T.

By way of comparison with the above results, the mechanical properties of the unalloyed titanium base metal as hot rolled below the beta transus temperature, vary somewhat with the amount of contaminants present, but in general fall within the following limits: 0.2% offset yield strength 65,000–80,000 p.s.i; ultimate strength 75,000–90,000 p.s.i.; tensile elongation 20–25%.

What is claimed is:

1. An alloy consisting essentially of from 1.0% to 5.0% manganese, from 1.0% to 2.0% iron, with the minimum iron plus manganese content being about 2.5 atomic percent, and the balance titanium, characterized by a tensile strength of at least about 25% in excess of the tensile strength of unalloyed titanium, measured in the as-hot-rolled (at about 1200° F.) condition.

2. An alloy consisting essentially of from about 3% to about 7.5% chromium, from about 0.2% to about 0.5% carbon, and the balance titanium, said alloy being characterized by having a minimum tensile strength of about 128,000 p.s.i. and a minimum elongation of about 5.0% in the as-hot-rolled (at about 1250° F.) condition.

3. An alloy consisting essentially of from about 2.5% to about 5% molybdenum, about 0.1% nitrogen, and the balance substantially titanium, said alloy being characterized by a minimum tensile strength of at least 130,000 p.s.i. and a minimum elongation of at least 2.5%

4. An alloy consisting essentially of from about 1% to about 5% manganese, about 0.1% nitrogen, from about 0.25% to about 0.5% carbon, and the balance substantially titanium, said alloy being characterized by a minimum tensile strength of at least 137,000 p.s.i. and minimum elongation of at least 2%.

5. An alloy consisting essentially of from about 2.0% to about 5.0% molybdenum, about 0.1% nitrogen, from about 0.25% to about 0.5% carbon, and the balance substantially titanium, said alloy being characterized by a minimum tensile strength of at least 143,000 p.s.i. and minimum elongation of at least 2%.

6. An alloy consisting essentially of from about 1.5% to about 7.5% manganese, about 0.1% nitrogen, and the balance substantially all titanium, said alloy being characterized by a minimum tensile strength of at least 134,400 p.s.i. and a minimum elongation of at least 2.0%.

7. A titanium-base alloy consisting essentially of about: 2.5 to 15 atomic percent of at least two beta promoting elements selected from the group consisting of manganese, chromium, molybdenum and iron, chromium when present with molybdenum alone, being not under 3% by weight, up to 0.5% carbon, up to 0.3% oxygen, up to 0.1% nitrogen, up to 8% aluminum, balance substantially titanium, characterized as plastically deformed in the alpha-beta temperature field, by tensile strength of at least 130,000 p.s.i. and a tensile elongation of at least 2%.

8. A titanium-base alloy consisting essentially of about: 2.5 to 15 atomic percent of at least three elements selected from the group consisting of manganese, chromium, molybdenum and iron, carbon up to 0.5%, oxygen up to 0.3%, nitrogen up to 0.1%, up to 8% aluminum, balance substantially titanium, characterized as plastically deformed in the alpha-beta temperature field, by a tensile strength of at least 130,000 p.s.i. and a tensile elongation of at least 2%.

9. A titanium-base alloy consisting essentially of about: 2.5 to 15 atomic percent of at least two elements selected from the group consisting of manganese, chromium, molybdenum and iron, 0.5 to 8% aluminum, up to 0.5% carbon, up to 0.3% oxygen, up to 0.1% nitrogen, balance substantially titanium, characterized by high strength and ductility.

10. A titanium-base alloy consisting essentially of about: 2.5 to 15 atomic percent of at least three elements selected from the group consisting of manganese, chromium, molybdenum and iron, 0.5 to 8% aluminum, up to 0.5% carbon, up to 0.3% oxygen, up to 0.1% nitrogen, balance substantially titanium, characterized by high strength and ductility.

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