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TITANIUM-NIOBIUM ALLOYS

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This invention relates to titanium-niobium-base alloys, particularly titanium rich and intermediate alloys containing up to 70 percent niobium.

This application is a continuation-in-part of our application Serial No. 575,937, filed April 4, 1956, now abandoned.

The binary titanium-niobium equilibrium diagram shows a beta-isomorphous system with a limited solubility of niobium in alpha titanium and a rather extensive alpha-beta field. At 1100° F., about 3 percent niobium can be dissolved in alpha, while the alpha-beta field extends from about 3 to 35 percent niobium. On quenching from the beta field, beta is completely retained at 36 percent niobium and above. The addition of a third element to Ti-Nb-base compositions provides further useful alloys.

It is an objective of this invention, therefore, to provide new and valuable titanium-niobium-base ternary alloys.

Still another object of this invention is to provide ternary alloys of titanium and niobium, and of titanium-niobium-base compositions, whereby valuable properties

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base ternary alloys containing up to 70 percent niobium, up to 45 percent of an alloying addition, balance titanium. Specific alloying elements of interest include vanadium, tantalum, and zirconium.

Binary alloys of titanium and niobium have been prepared by melting in an inert tungsten-electrode arc furnace under a static argon partial pressure of 15 to 20 centimeters of mercury. Alloy compositions of 1.5 percent, 3 percent, 10 percent, 15 percent, 20 percent, 25 percent, 30 percent, 35 percent, 40 percent, 50 percent, 60 percent, and 70 percent niobium, balance essentially all titanium have been prepared. Except for some spatter during the melting of the 60 percent and 70 percent niobium alloys, no difficulties have been encountered in preparing these compositions. Alloy specimens containing up to 70 percent niobium have been forged in air to a 3/8-inch-square cross section. The forged ingots have been rolled in air at 1500° F. to approximately 0.040-inch sheet, using 10 percent reduction per pass. The alloys may then be given an annealing or stabilizing treatment.

Ternary titanium-niobium-base alloys have also been prepared by melting in an inert tungsten-electrode arc furnace under a static argon partial pressure of 15 to 20 centimeters. Table I lists mechanical properties of some ternary alpha-beta-type alloys. Data for binary Ti-10Nb and Ti-15Nb compositions are included for comparison. The worked ternary alloys were all in the equilibrated-and-stabilized condition, i.e., held 1/2 hour at 1300° F., furnace cooled to 1100° F., held 1 hour and air-cooled.

Table I.—Mechanical Properties of Ti-Nb-Base Ternary Alpha-Beta Alloys

Composition Weight percent (balance Ti)	Vickers hardness Number 10-kg. load		Tensile strength (p.s.i.)	Yield strength, p.s.i.		Elongation, per- cent in 1-inch		Reduction in area, percent	Bend ductility
	As-cast	Wrought		0.1% offset	0.2% offset	Uni- form	Total		
10 Nb.....	205	191	71,000	54,000	58,000	10	18	47	0
15 Nb.....	242	197	74,000	54,000	60,000	8	13	33	0
10 Nb-5 V.....	299	228	88,000	77,000	81,000	5	7	26	0
10 Nb-5 Ta.....	233	207	81,000	65,000	70,000	6	10	32	0
10 Nb-5 Zr.....	230	215	98,000	77,000	82,000	5	10	23	0.5

of heat-treatability and useful strength-ductility relationships may be obtained and/or combined.

Yet another object of this invention is to provide effective titanium-niobium-base ternary compositions, with tantalum, zirconium, or vanadium as the third component.

Other objects and advantages of the present invention will be apparent from the following examples and detailed descriptions thereof.

In general, this invention relates to titanium-niobium-

An investigation of the aging properties of the alpha-beta alloys has shown that alloys containing 10 percent niobium and 5 percent of another beta-stabilizing element have very high aging responses. In this investigation the ternary alpha-beta alloys were quenched from 1250° F. and 1550° F. and then aged at 800° F. for periods of time up to 16 hours. Alloys containing vanadium harden appreciably on quenching from either 1250° F. or 1550° F. Table II below shows the results of the aging studies in terms of hardness.

Table II.—Aging Response of Ternary Ti—Nb—Base Alpha-Beta Alloys Aged at 800° F.

Alloy weight percent addition	Aging time, hours	Quench	
		VHN(10-kg.) ^a	VHN(10-kg.) ^b
Ti-10 Nb-5 V.....	0	317	239
	1/4	333	262
	1	317	306
	2	309	333
	4	319	319
Ti-10 Nb-5 Ta.....	16	260	354
	0	236	215
	1/4	254	210
	1	260	215
	2	245	225
Ti-10 Nb-5 Zr.....	4	254	247
	16	235	228
	0	240	240
	1/4	251	249
	1	242	276
	2	233	236
	4	242	249
	16	230	260

^a Specimens held at 1,250° F. for 4 hours and water quenched prior to aging.

^b Specimens held at 1,550° F. for 1 hour and water quenched prior to aging.

These investigations indicate the value of alpha-beta-type alloys containing from about 3 percent to about 30 percent niobium, from about 1 percent to about 10 percent of at least one of the alloying elements zirconium, tantalum, vanadium, balance essentially titanium. The data illustrate the useful properties of this alloy system.

In addition to the alpha-beta-type alloys discussed above, it has been found that titanium-niobium-base ternary alloys of the beta type possess valuable properties and useful characteristics. Table III below lists the mechanical properties of these Ti—Nb-base ternary alloys.

Table III.—Mechanical Properties of Ti—Nb—Base Ternary Beta Alloys

Composition weight percent (balance Ti)	Vickers hardness number 10-kg. load		Tensile strength (p.s.i.)	Yield strength, p.s.i.		Elongation, percent in 1 inch		Reduction in area, percent	Bend ductility
	As-cast	Wrought ^a		0.1% Offset	0.2% Offset	Uniform	Total		
50 Nb.....	153	138	58,000	58,000	58,000	0	0	51	0
50 Nb-5 V.....	161	164	72,000	72,000	72,000	10	17	47	0
50 Nb-10 V.....	219	199	83,000	80,000	80,000	13	17	42	0
50 Nb-20 V.....	266	272	106,000	98,000	98,000	13	18	40	0
50 Nb-30 V.....	249	283	126,000	118,000	119,000	13	17	48	0
50 Nb-40 V.....	285	304	141,000	134,000	135,000	3	3	8	1.0
50 Nb-50 V.....	360	390							
50 Nb-5 Ta.....	177	140	63,000	63,000	63,000	0	16	41	0
50 Nb-10 Ta.....	181	149	66,000	65,000	66,000	0	16	54	0
50 Nb-20 Ta.....	202	179	74,000	74,000	74,000	12	19	54	0
50 Nb-30 Ta.....	207	206							
50 Nb-40 Ta.....	206	224							
50 Nb-50 Ta.....	169	233							
50 Nb-10 Zr.....	215	167	780,000	77,000	77,000	8	13	39	0
50 Nb-20 Zr.....	245	209	100,000	95,000	96,000	9	13	40	0
50 Nb-30 Zr.....	262	249	114,000	110,000	110,000	13	20	55	0
50 Nb-40 Zr.....	268	276							
50 Nb-50 Zr.....	272	272							

^a Samples held 2 hours at 1,450° F. and water quenched.

Mechanical-property data for the Ti—50Nb binary composition are included in the table for purposes of comparison. It should be noted that the ternary additions strengthened the base material. These alloys also show considerable increase in uniform elongation over the binary 50 percent niobium composition.

Hot-hardness data for the alloys are presented in Table IV. Values are tabulated for 75° F., 930° F., 1290° F., and 1650° F.

At 930° F. and 1290° F. vanadium gives the most effective strengthening per unit ternary addition, followed in order by zirconium and tantalum. However at 1650° F. the order is zirconium, vanadium and tantalum. At 20 percent addition and above, the ternary additions vanadium, zirconium, and tantalum strengthen much more, proportionately, at the higher temperatures than

they do at the lower temperatures. Thus, they provide useful high temperature alloys. Additions of from about 5 percent to about 45 percent of the elements to titanium-niobium alloys containing 30 to 70 percent niobium are beneficial. Outstanding hot hardness properties are obtained by adding 30 to 40 percent of vanadium or zirconium to titanium-niobium alloys containing about 40 to 60 percent niobium.

Other alloy systems disclosed possess extremely valuable properties. For example Ti—50Nb-base alloys containing vanadium and zirconium have excellent room-temperature ductility, very good uniform elongation, and improved strength at 1650° F. It is clear also that complex alloys containing combinations of the individual alloying elements will possess equally important and worthwhile properties.

Table IV.—Hot Hardness of Ti—Nb Base Alloys

Composition, weight percent (balance Ti)	Hardness, ^a DPH, kg./mm. ² , at temperature ° F. indicated			
	75	930	1,290	1,650
50 Nb.....	138	94	40	19
60 Nb.....	146	107	66	40
70 Nb.....	159	96	79	58
50 Nb-5 V.....	164	177	78	37
50 Nb-10 B.....	199	162	100	42
50 Nb-20 V.....	272	217	139	74
50 Nb-30 V.....	283	229	194	141
50 Nb-40 V.....	304	282	242	178
50 Nb-5 Ta.....	140	83	46	25
50 Nb-10 Ta.....	149	87	52	27
50 Nb-20 Ta.....	179	152	113	99
50 Nb-30 Zr.....	167	149	89	47
50 Nb-40 Zr.....	209	179	119	70
50 Nb-50 Zr.....	249	198	149	121

^a Elevated-temperature tests were conducted in a vacuum of less than 5 microns, using a load of 730 grams and an indentation time of 10 seconds

The data in Table V below show the results of tests to determine the resistance of the Ti—Nb base ternary alloys to oxidation in a static atmosphere at elevated temperatures. Specimens used in obtaining these data were about 0.25 x 0.50 x 0.04 inch and were prepared as follows: Each specimen was carefully finished on 400-grit paper, cleaned in a solvent, weighed and measured. Each specimen was then placed on a small ceramic ring and set inside a crucible cover. A second, larger ceramic ring was then placed around each specimen to insure retention of any spattered scale.

Each specimen assembly, prepared as described above, was placed on a firebrick and, thus supported, inserted into a furnace which had been brought up to the test temperature. The specimens were in this manner exposed to air oxidation for periods of 16 hours at test tem-

peratures of 1400° F. and 1800° F. After exposure for the prescribed time, the treated specimens were covered with a second crucible cover and cooled. In this way, the loss of oxidation products during the cooling period was avoided.

After the specimens had been cooled, they were each weighed to determine the weight gain of each specimen. From this, the weight increase per unit original area was computed for each specimen. The samples were then brushed with a wire brush to remove loose scale, and the percentage of original sample remaining was then determined. In many cases the scale proved to be tightly adherent and could not be removed entirely, which accounts for the fact that values greater than 100 are reported in several instances under "percent of original sample remaining." The samples were then sectioned centrally, mounted in Bakelite, polished, and etched for microexamination. The thickness of unaffected base material was measured by means of a calibrated movable stage. Finally, hardness readings were taken in the center of the cross section. Values obtained in accordance with the procedures described above were as follows:

Table V.—Resistance of Ti—Nb Base Alloys to Oxidation in a Static Atmosphere

Composition weight percent (balance Ti)	Original Vickers hardness (10-kg. load)	16 hours at 1,400° F.				16 hours at 1,800° F.			
		Weight increase per unit original area, mg./sq. cm.	Penetration of contaminant per side, inch	Percent of original sample remaining	Vickers hardness (10-kg. load)	Weight increase per unit original area, mg./sq. cm.	Penetration of contaminant per side, inch	Percent of original sample remaining	Vickers hardness (10-kg. load)
50 Nb.....	138	2.58	0.0063	101	202	73.7	Total	18	370
60 Nb.....	146	3.54	0.0063	101	155	61.5	Total	46	380
70 Nb.....	159	6.96	0.0052	97	156	92.4	Total	16	-----
50 Nb-5 V.....	164	3.90	0.0065	101	180	52.9	Total	0	-----
50 Nb-10 V.....	199	7.20	0.0057	103	215	72.6	Total	0	-----
50 Nb-20 V.....	272	14.17	0.0040	103	238	126.9	Total	0	-----
50 Nb-30 V.....	283	172.6	Total	0	-----	171.0	Total	0	-----
50 Nb-40 V.....	304	108.4	Total	0	-----	108.5	Total	0	-----
50 Nb-5 Ta.....	140	2.86	0.0063	101	210	63.4	Total	37	376
50 Nb-10 Ta.....	149	3.25	0.0060	101	170	62.6	Total	57	409
50 Nb-20 Ta.....	179	4.67	0.0040	101	170	65.2	Total	29	380
50 Nb-10 Zr.....	167	4.31	0.0033	102	357	72.0	Total	10	-----
50 Nb-20 Zr.....	209	17.44	0.0068	98	276	69.9	Total	0	-----
50 Nb-30 Zr.....	249	112.1	Total	0	-----	73.0	Total	0	-----

From the values listed above it is apparent that 16 hours' exposure at 1400° F. had no unduly severe effects on most of the alloys. Except for the Ti—50 Nb—20 Zr alloy, which flaked slightly, a tightly adherent scale was formed on all the specimens. As has been noted above, this scale could not always be entirely removed by a wire brush, and consequently many of the values for "percent of original sample remaining" are in excess of 100.

The alloys of this invention will find many applications. They are particularly useful for structural members for high temperature applications. They are useful for mis-

siles, for jet engine parts and for such specific uses as leading edge wing sections of high speed aircraft for upper atmosphere operation.

New and useful alloys of titanium and niobium and of titanium-niobium-base alloys having been described and characterized, it is desired to further define the invention according to the following claims.

What is claimed is:

1. Titanium-niobium alloys consisting of from about 40 percent to about 60 percent niobium, from about 30 percent to about 40 percent vanadium, balance titanium, said alloys being characterized by good hot hardness properties.

2. Titanium-niobium alloys consisting of from about 40 percent to about 60 percent niobium, from about 30 percent to about 40 percent zirconium, balance titanium, said alloys being characterized by good hot hardness properties.

3. Titanium-niobium-base alloys of the beta-phase type consisting of from about 30 percent to about 70 percent niobium, from about 30 percent to about 40 percent of an element from the group consisting of vanadium, zirconium, and tantalum, balance titanium.

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**UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION**

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, Table II, column 4, line 1 thereof, for "239" read -- 230 --; columns 3 and 4, Table III, column 4, line 14 thereof, for "780,000" read -- 78,000 --.

Signed and sealed this 16th day of October 1962.

(SEAL)

Attest:

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