

1

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NIOBIUM BASE ALLOY

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This invention relates to niobium-base alloys, and more particularly to niobium-base alloys containing vanadium and zirconium and preferably one element selected from the group consisting of aluminum and iron.

The rapid growth of the airframe and missile technologies, for example, has made necessary a new appraisal of possible high temperature alloys. The present materials based on iron, cobalt or nickel are limited, in general, to a 1500° F. temperature ceiling. While molybdenum alloys show great promise at elevated temperatures, the density and fabricability of niobium makes it of interest for structural members.

Accordingly, it is a principal object of the invention to provide improved niobium-base alloys for high temperature service.

Another object of the invention is to provide alloys of the above type which possess good workability and high temperature oxidation resistance.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description.

Therefore, in accordance with the present invention, it has been found that castable and forgeable alloys possessing exceptionally high oxidation resistance and strength at temperatures above 1000° C. can be produced by alloying niobium, vanadium, zirconium and specified added elements set forth herein.

The improved alloys of the present invention comprise compositions containing between about 8 to 20% vanadium, 0.1 to 5% zirconium, and 0 to 2% aluminum or iron, the sum total of C, O and N ranging from 0.1 to 0.2% and the balance being essentially niobium. It was found that when up to 20% titanium was added the already high oxidation resistance was further increased.

In a more preferred embodiment of this invention the alloys comprise between about 12% vanadium, 2% zirconium, 1% aluminum or ½% iron, the sum total of C, O and N ranging from 0.1 to 0.2%, the balance being essentially niobium.

The alloys of this invention are prepared according to the following procedure employing conventional techniques. For melting operations, the alloying elements were reduced in size when necessary by successive passages through a "Wiley" mill so that the maximum particle size was less than 20 mesh. Following comminution, all of the metal was cleaned by magnetic separation and, where practical, degassed by vacuum sintering. Measured amounts of the individual metal powder constituents were thoroughly mixed, using a chemical riffle and recycling to insure uniformity. The mixed material was then pressed into bars 24 inches long by 1¼ inches in diameter at pressures of approximately 20 tons per square inch. Two of such bars were heliarc welded end to end. Following welding, the bars were degassed and sintered by heating them resistively in a vacuum furnace to approximately 1500° C. while maintaining a vacuum of 10⁻⁴ mm. Hg abs. The bars were then consumably arc melted into a cold mold at ½ atmosphere of argon. The melting operation is not limited to arc melting under argon but can also be carried out in vacuum and electron beam melting can be utilized in place of arc melting.

Alloys prepared according to the above method and

2

falling within the scope of this invention are found to possess the following composition:

(I)

	Percent
5 Vanadium -----	8 to 20
Titanium -----	0 to 20
Zirconium -----	0.1 to 5
Aluminum or iron -----	0 to 2
C, O, N -----	0.1 to 0.2
10 Niobium -----	Balance

(II)

Vanadium -----	8 to 20
Zirconium -----	0.1 to 5
15 Titanium -----	10 to 20
Iron or aluminum -----	0.1 to 2
Niobium -----	Balance

(III)

Vanadium -----	12
20 Zirconium -----	2
Iron -----	0.5
C, O, N -----	0.1 to 0.2
Niobium -----	Balance

(IV)

25 Vanadium -----	12
Zirconium -----	2
Aluminum -----	0.5 to 1
C, O, N -----	0.1 to 0.2
30 Niobium -----	Balance

Typical improved high temperature properties of preferred niobium-base alloy may be illustrated by the following tests on the above mentioned alloys designated as (III) and (IV).

Dynamic hot stiffness tests

As an index of the deformation resistance during fabrication or use, a dynamic hot stiffness test was applied to small pieces of the alloys. Rectangular pieces of equal size and shape were cut and the sides ground flat and parallel. No rounding of the edges was permitted. Each pellet was measured as to length, width, and height before deformation and the final thickness after the test was also measured. One blow of 2700 pound inches given by dropping a 600 lb. hammer once through a distance of 4½ inches was applied to each pellet which was at a temperature of 2000° F. The average deformation was approximately 32%.

Static hot hardness test

These tests were carried out in a conventional hot hardness machine. The work was supported on a heavy molybdenum anvil in which the thermo-couple is buried. Radiation shields surround the hot zone. A molybdenum extension bar connects the indenter to the Rockwell hardness machine through a bellows arrangement. A carefully calibrated spring corrects the effects of air pressure in compressing the water-cooled bellows. The hot hardness runs were made at pressures of less than 5×10⁻⁶ mm. Hg abs. at operating temperature. The Rockwell "A" hardness remained between 60 and 70 for temperatures up to 2300° F. In most cases not over a 5 point Rockwell "A" drop was found for samples held at 2000° F. for fifteen minutes.

Hot tensile tests

The alloy specimens whose gauge length was approximately 1 inch long by 0.200, by 0.075 thick were placed in a tensile testing machine. The temperature was recorded on a standard chart recorder. The tensile tests were run at 0.05 inch per minute crosshead travel. In the as-cast condition, the strength in tension was approxi-

mately 70,000 to 82,000 p.s.i. or better at 2000° F. In comparison, the highest reported ultimate tensile strength obtained for a molybdenum 0.5 percent titanium alloy was 67,500 p.s.i. at 1800° F. on material forged at 2100° F. in the as-rolled state. This molybdenum alloy is stated to be the strongest available for test.

Still air oxidation resistance testing

In this test, the alloy specimens were carefully cleaned and lightly surface etched. After estimating the surface area, the specimens were weighed and placed in baked-out porcelain crucibles and held for one half hour at 1000° C. in still undried air. Following exposure, the crucibles were weighed and put back into the furnace for an additional one half hour. After this additional treatment, the specimens were reweighed. The weight gain was approximately 7 milligrams per square centimeter for the first one half hour and the total weight gain in one hour was 12 mgm./cm.².

It is to be noted that, although it is preferable to use metals of high purity, those skilled in the art are readily aware of incidental impurities in commercial metals, and this fact should be taken into consideration when practicing the invention and construing the claims.

Thus it can be seen that the improved high temperature

properties of the alloys of the present invention make them especially suitable for various high temperature services.

Since certain changes may be made in the above described details without departing from the scope of the invention herein involved, it is intended that all matter contained in the description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. An improved high temperature service niobium base and alloy comprising by weight approximately of 12% vanadium, 2% zirconium, 0.5% iron, the sum total of C, O and N ranging from 0.1 to 0.2%, the balance being essentially niobium.

2. An improved high temperature service niobium base alloy comprising by weight approximately of 12% vanadium, 2% zirconium, 0.5 to 1% aluminum, the sum total of C, O and N ranging from 0.1 to 0.2%, the balance being essentially niobium.

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