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METHOD OF MAKING COLD WORKED AND AGED PRODUCTS WHICH ARE SUBSTANTIALLY FREE OF OBJECTIONABLE LAMELLAR CONSTITUENT FROM PRECIPITATION HARDENABLE FERROUS BASE ALLOYS

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This invention relates to a method of making cold worked and aged products which are substantially free of objectionable lamellar constituent from precipitation hardenable ferrous base alloys, which alloys have high strength at elevated temperatures. Alloys of the type referred to are used for parts in jet engines for aircraft, gas turbines and other high temperature applications where it is necessary to have consistently high strength properties. The specifications for these alloys usually require that after the alloy is solution heat treated at 1650–1800° F. for a time sufficient to dissolve the intermetallic compounds (usually for 1 to 2 hours), cooled to room temperature by quenching in air, oil or water or any other medium sufficient to prevent any precipitation of an intermetallic compound of an element which has been taken into solution at the solution heat treatment temperature, aged at 1300–1400° F. for a time sufficient to obtain the desired hardness (usually 16 hours at 1300–1325° F.) and air cooled to room temperature, the alloy must have a minimum stress rupture life of 23 hours when tested at a temperature of 1200° F. and a load of 65,000 p.s.i.

The term "quenched" as used herein means cooling the solution heat treated alloy to room temperature at a rate sufficiently rapid to prevent any precipitation of an intermetallic compound which has been taken into solution at the solution heat treatment temperature.

This application is a continuation-in-part of my application Serial No. 729,164, filed April 17, 1958, now abandoned.

The alloys to which the present invention relates consist essentially of the following elements in the proportions stated, the balance being iron:

	Percent
Ni	24–28
Cr	12–16
Ti	1.35–4.5
B	Up to 0.15
C	Up to 0.20
Al	Up to 2.0
Mn	Up to 3.0
Si	Up to 2.0
Mo	Up to 5.0
V	Up to 1.0
Zr	Uu to 0.5
Cb+Ta	Up to 2.0

The ranges of elements for three alloys coming within the present invention are as follows:

TABLE I

	Alloy No. 1	Alloy No. 2	Alloy No. 3
Ni	24.0–27.0	24.0–28.0	24.0–27.0
Cr	13.5–16.0	12.0–15.0	13.5–16.0
Ti	1.75–3.00	1.35–3.0	3.5–4.5
B	0.0006–0.15	0.0006–0.15	0.0006–0.15
C	0.08 max.	0.08 max.	0.10 max.
Al	Up to 0.35	Up to 0.35	Up to 0.50
Mn	0–2.0	0–1.0	0–2.0
Si	0–1.0	0–1.0	0–1.0
Mo	1.0–1.5	2.5–3.5	------------
V	0.10–0.5	------------	------------
Zr	------------	------------	0.01–0.15
Cb+Ta	------------	------------	0.45–1.0

A typical preferred alloy has the following composition:

ALLOY No. 4

	Percent
Ni	25.00
Cr	14.75
Ti	2.00
B	0.003
C	0.06
Al	0.25
Mn	1.50
Si	0.68
Mo	1.26
V	0.25
Fe	Balance

Although the boron content of my alloys can be up to 0.15%, the preferred range is from 0.001 to 0.05%.

Alloys of the type hereinabove referred to have a satisfactory stress rupture life when they are solution heat treated at a temperature of 1650–1800° F., quenched and aged at a temperature of 1300–1400° F. For example, an alloy of the composition of Alloy No. 4, when solution heat treated at a temperature of 1800° F. for 1 hour, quenched and then aged at a temperature of 1325° F. for 16 hours, has a stress rupture life of about 100 hours when tested at 1200° F. under a load of 65,000 p.s.i. In further reference to stress rupture life, it will be understood that it is the stress rupture life when tested at 1200° F. under a load of 65,000 p.s.i. which is referred to unless some other conditions are stated.

From the above, it is evident that Alloy No. 4 and the other alloys previously referred to herein are entirely satisfactory where the treatment of the alloy involves only solution heat treating and quenching followed by an aging treatment. However, there are applications of the alloy where it is necessary to severely cold work it between the solution heat treating and aging steps. The best example of this application is the manufacture of bolts in which the cold headed blank from which the bolt is to be made is solution heat treated at a temperature of 1650–1800° F., quenched, thread rolled in the cold state and then aged at a temperature of 1300–1400° F. Bolts made from Alloy No. 4 by solution heat treating at 1800° F. for 1 hour, quenching, thread rolling in the cold state and then aging at 1325° F. for 16 hours showed a lamellar precipitate or constituent in the severely cold worked threads, the lamellar precipitate being very similar in appearance to the pearlite commonly noted in carbon steels.

Bolts made in this manner, although having a stress rupture life of over 23 hours, and thus meeting the specification requirements, did not approach the stress rupture life of bars made from the same alloy which had been solution heat treated, quenched and aged in the same manner but without being cold worked between the solution heat treatment and aging treatment. The stress rupture life of the thread rolled material was about 24 hours as compared with the stress rupture life of about 100 hours for the product which had been solution heat treated, quenched and aged without an intervening cold thread rolling operation.

An object of the invention is to provide a heat treatment for alloys of the type hereinabove referred to which will prevent or substantially reduce the amount of lamellar constituent which is normally formed when the alloy is solution heat treated at a temperature of 1650–1800° F., quenched and then subjected to severe cold working, for instance, thread rolling in the cold state, and thereafter aged at a temperature of 1300–1400° F.

Another object of the present invention is to provide a heat treatment which will eliminate or substantially reduce the lamellar constituent in alloys of the type referred to, which alloys have been solution heat treated at a temperature of 1650–1800° F., quenched and then subjected to severe cold working, for instance, thread rolling in the cold state, and then aged at a temperature of 1300–1400° F.

In one embodiment of my invention, the alloy is treated in the following manner:

(*a*) It is solution heat treated at a temperature of 1650–1800° F. for a time sufficient to dissolve the intermetallic compounds present and is then quenched.

(*b*) It is then cold worked.

(*c*) It is then given a high temperature solution heat treatment at a temperature of 1950–2200° F. for a time sufficient to obtain solution of the intermetallic compounds and is then quenched.

(*d*) It is aged at a temperature of 1300–1400° F. for a time sufficient to obtain the desired hardness. The aging causes precipitation of intermetallic compounds.

A preferred example of this embodiment of the invention is as follows, the alloy being of the composition of Alloy No. 4.

Example 1

(*a*) The alloy was solution heat treated for 1 hour at 1800° F. and quenched in oil.

(*b*) It was then thread rolled in the cold state.

(*c*) The thread rolled product was given a high temperature solution heat treatment by holding it for 1 hour at 2000° F. and was then quenched in oil.

(*d*) It was aged at 1325° F. for 16 hours.

The product resulting from Example 1 did not show any substantial amount oft he lamellar constituent. This material had a stress rupture life of 51 hours.

In Example 1, the high temperature solution heat treatment, step (c), prevents the formation of any substantial amount of lamellar constituent when the alloy is aged in step (*d*). Thus, the high temperature solution heat treatment is a preventive measure for preventing the formation of the objectionable lamellar constituent upon aging.

The present invention also is applicable to eliminating or decreasing the amount of lamellar constituent resulting from solution heat treating the alloy, quenching it, severely cold working it and then aging it. In this embodiment, the series of steps is as follows:

(*a*) The alloy is solution heat treated at 1650–1800° F. to dissolve the intermetallic compounds and is quenched.

(*b*) It is subjected to severe cold working.

(*c*) It is aged at 1300–1400° F.

This series of steps (*a*), (*b*) and (*c*) results in a product having a lamellar constituent and a relatively low stress rupture life. The remedial steps according to my invention for eliminating or decreasing the lamellar constituent consist of the following steps (*d*) and (*e*).

(*d*) The product is given a high temperature solution heat treatment at 1950–2200° F. for a time sufficient to dissolve the intermetallic compounds and cause the disappearance of the lamellar constituent and is quenched.

(*e*) It is then aged at 1300–1400° F. for a sufficient time to obtain the desired hardness.

The following Example 2 further illustrates this embodiment which relates to the remedial treatment.

Example 2

(*a*) An alloy of the composition of Alloy No. 4 was solution heat treated at 1800° F. for 1 hour and quenched in oil.

(*b*) It was then thread rolled in the cold state.

(*c*) It was aged at 1325° F. for 16 hours.

(*d*) It was then given a high temperature solution heat treatment at 2000° F. for 1 hour which dissolved the intermetallic compounds and caused the disappearance of the lamellar constituent and was quenched in oil.

(*e*) It was aged at 1325° F. for 16 hours.

The product did not contain any substantial amount of lamellar constituent. This material had a stress rupture life of 36 hours.

In accordance with my invention, the sequence of the normal solution heat treatment (1650–1800° F.), severe cold working and high temperature solution heat treatment (1950–2200° F.) is important. Thus, the high temperature solution heat treatment will not accomplish the desired results if it is employed in the wrong sequence relative to the cold working or the normal solution heat treatment. For instance, a high temperature solution heat treatment which precedes the cold working and aging does not produce the desired results. The following sequence is unsatisfactory.

Example 3

(*a*) An alloy of the composition of Alloy No. 4 was solution heat treated at 2000° F. for 1 hour and quenched in oil.

(*b*) It was thread rolled in the cold state.

(*c*) It was aged at 1325° F. for 16 hours.

The product contained a substantial amount of lamellar constituent.

In accordance with my invention, the high temperature solution heat treatment (1950–2200° F.) must follow the cold working (Example 1) or must follow the aging (Example 2). In other words, a severe cold working must not intervene between the high temperature solution heat treatment and the aging treatment (Example 3).

Another point to be noted is that the aging treatment at 1300–1400° F. must follow the high temperature solution heat treatment (1950–2200° F.) without there being a solution heat treating step at a temperature substantially lower than 1950° F. intervening between the high temperature solution heat treatment and the aging treatment. This is illustrated by the following example.

Example 4

(*a*) An alloy of the composition of Alloy No. 4 was given a high temperature solution heat treatment at 2000° F. for 1 hour and quenched in oil.

(*b*) It was then thread rolled in the cold state.

(*c*) It was aged at 1325° F. for 16 hours.

This resulted in a lamellar constituent in the product.

(*d*) The product was solution heat treated at 1800° F. for 1 hour which dissolved the intermetallic compounds and caused the disappearance of the lamellar constituent, and the product was quenched in oil.

(e) The product was aged at 1325° F. for 16 hours whereupon the lamellar constituent reformed.

A preferred method of making a bolt is given in the following example.

Example 5

(a) An alloy bar of the composition of Alloy No. 4 was solution heat treated at 1800° F. for 1 hour and quenched in oil.

(b) The bar was cold headed.

(c) It was solution heat treated at 1650° F. for 1 hour and quenched in oil.

(d) It was thread rolled in the cold state.

(e) It was solution heat treated at 2000° F. for 1 hour and quenched in oil.

(f) It was aged at 1325° F. for 16 hours.

While, according to my invention, the high temperature solution heat treatment range is from 1950–2200° F., it is preferred to employ temperatures between 2000° F. and 2200° F.

The invention is not limited to the preferred embodiment but may be otherwise embodied and practiced within the scope of the following claims.

I claim:

1. The method of making cold worked and aged products which are substantially free of objectionable lamellar constituent from precipitation hardenable ferrous base alloys consisting essentially of 24 to 28% nickel, 12 to 16% chromium, 1.35 to 4.5% titanium, up to 0.15% boron, up to 0.20% carbon, up to 2.0% aluminum, up to 3.0% manganese, up to 2.0% silicon, up to 5.0% molybdenum, up to 1.0% vanadium, up to 0.5% zirconium and up to 2.0% columbium plus tantalum, the balance being iron, which comprises solution heat treating said alloy at a temperature of 1650–1800° F., quenching it, cold working it, solution heat treating it at a temperature of 1950–2200° F., quenching it, and aging it at a temperature of 1300–1400° F.

2. The method of making cold worked and aged products which are substantially free of objectionable lamellar constituents from precipitation hardenable ferrous base alloys consisting essentially of 24 to 27% nickel, 13.5 to 16.0% chromium, 1.75 to 3.00% titanium, 0.0006 to 0.15% boron, up to 0.08% carbon, up to 0.35% aluminum, up to 2.0% manganese, up to 1.0% silicon, from 1.0 to 1.5% molybdenum and from 0.10 to 0.5% vanadium, the balance being iron, which comprises solution heat treating said alloy at a temperature of 1650–1800° F., quenching it, thread rolling it while cold, solution heat treating it at a temperature of 1950–2200° F., quenching it, and aging it at a temperature of 1300–1400° F.

3. The method of making cold worked and aged products which are substantially free of objectionable lamellar constituents from precipitation hardenable ferrous base alloys consisting essentially of 24 to 28% nickel, 12 to 16% chromium, 1.35 to 4.5% titanium, up to 0.15% boron, up to 0.20% carbon, up to 2.0% aluminum, up to 3.0% manganese, up to 0.5% zirconium and up to 2.0% columbium plus tantalum, the balance being iron, which comprises solution heat treating said alloy at a temperature of 1650–1800° F., quenching it, thread rolling it while cold, solution heat treating it at a temperature of 1950–2200° F., quenching it, and aging it at a temperature of 1300–1400° F.

4. The method of making cold worked and aged products which are substantially free of objectionable lamellar constituent from precipitation hardenable ferrous base alloys consisting essentially of 24 to 27% nickel, 13.5 to 16.0% chromium, 1.75 to 3.00% titanium, 0.0006 to 0.15% boron, up to 0.08% carbon, up to 0.35% aluminum, up to 2.0% manganese, up to 1.0% silicon, from 1.0 to 1.5% molybdenum and from 0.10 to 0.5% vanadium, the balance being iron, which comprises solution heat treating said alloy at a temperature of about 1650–1800° F., quenching it, thread rolling it while cold, solution heat treating it at a temperature of 1950–2200° F., quenching it, and aging it at a temperature of about 1325° F.

5. The method of eliminating objectionable lamellar constituent from precipitation hardenable ferrous base alloys consisting essentially of 24 to 28% nickel, 12 to 16% chromium, 1.35 to 4.5% titanium, up to 0.15% boron, up to 0.20% carbon, up to 2.0% aluminum, up to 3.0% manganese, up to 2.0% silicon, up to 5.0% molybdenum, up to 1.0% vanadium, up to 0.5% zirconium and up to 2.0% columbium plus tantalum, the balance being iron, said lamellar constituent resulting from solution heat treating said alloy at a temperature of 1650–1800° F., quenching it, cold working it, and aging it at a temperature of 1300–1400° F., which comprises solution heat treating it at a temperature of 1950–2200° F., quenching it, and aging it at a temperature of 1300–1400° F.

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