

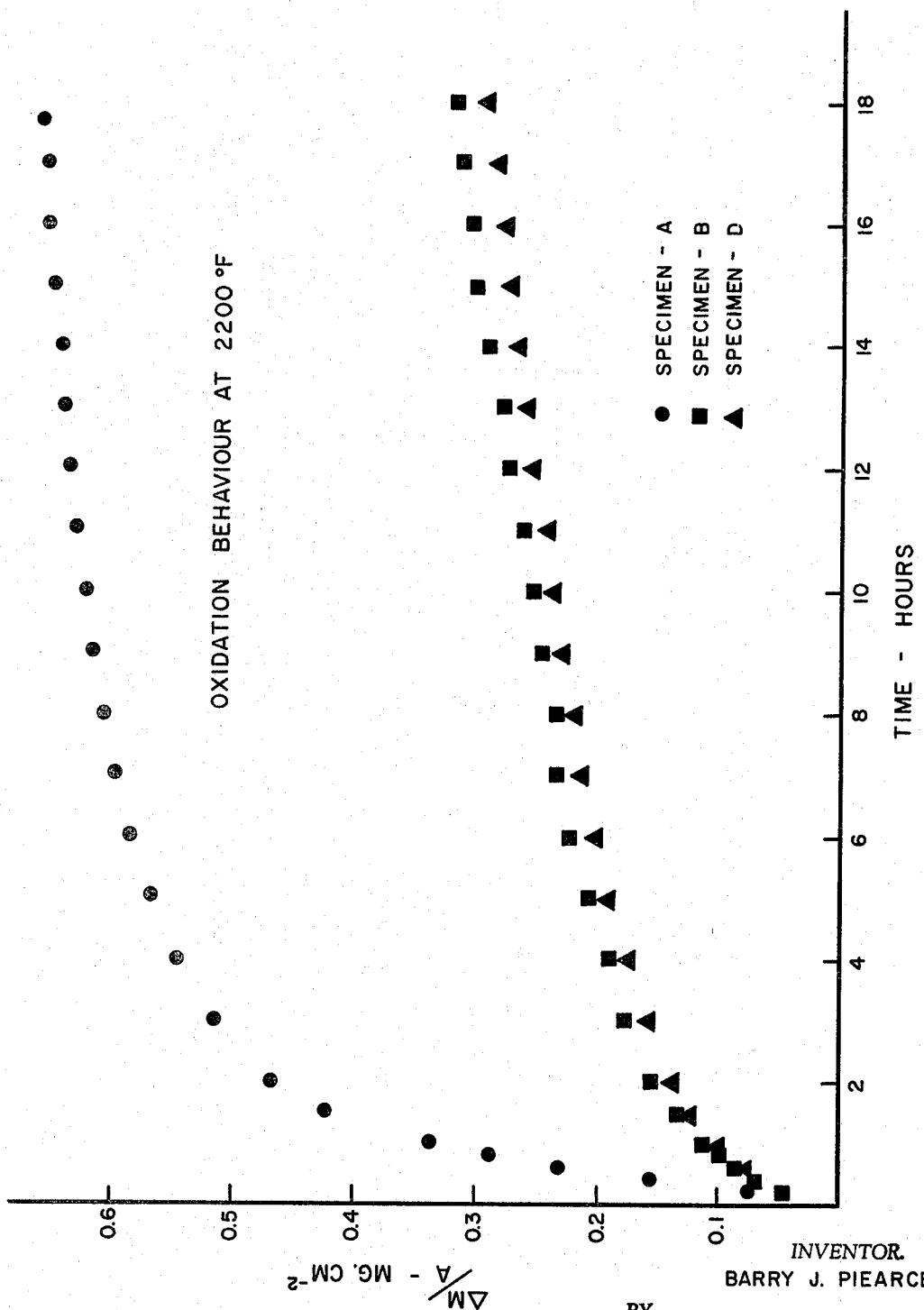
March 21, 1967

B. J. PEARCEY

3,310,440

HEAT TREATMENT OF NICKEL BASE ALLOYS

Filed Oct. 21, 1964



INVENTOR  
BARRY J. PEARCEY

BY  
MORGAN, FINNEGAN, DURHAM & PINE

ATTORNEYS

# United States Patent Office

3,310,440

Patented Mar. 21, 1967

1

3,310,440

## HEAT TREATMENT OF NICKEL BASE ALLOYS

Barry J. Piearcey, Cheshire, Conn., assignor to United Aircraft Corporation, East Hartford, Conn., a corporation of Delaware

Filed Oct. 21, 1964, Ser. No. 405,410  
5 Claims. (Cl. 148—13)

The present invention relates to the heat-treatment of nickel-base superalloys which are commonly coated to improve their high temperature oxidation, sulfidation and erosion resistance, which heat treatment improves the mechanical properties of the alloys compared with their normal properties in the coated condition.

Heretofore, nickel-base alloys for use in gas turbine power plants, either as rotor or stator members have been provided with coatings adapted to render the blades or vanes more highly resistant to oxidation and thermal shock. However, the treatment which provides the resistant coating tends somewhat to reduce the blade life under stress at intermediate high temperatures and to reduce the elongation of such blades.

The present invention has for its object the provision of a novel heat treatment of the blade or vane members formed from nickel-base superalloys, such as those used for the production of gas-turbine blade and vane members for use at temperatures of 1200° F. up to much higher temperatures, so as to greatly improve their life in use, while not impairing their other desirable properties.

A further object is the provision of a novel and improved process for the heat treatment of certain cast nickel-base superalloys so as to improve the creep properties of such alloy members at intermediate and elevated temperatures.

The process of the present invention is particularly adapted for use with the nickel-base, coated superalloys which are used in gas turbine blades and vanes to be operated at temperatures in excess of 1200° F. and even as high as 2200° F. under extreme conditions. Such blades are conventionally provided with an adherent surface coating which contributes greatly to the improvement of the properties of the blades and vanes with respect to high temperature oxidation, sulfidation and erosion resistance, but often does so at the cost of some reduction in the life of such blades under continuously applied stress such as 1400° F. and 95,000 p.s.i.

As the result of the present invention, gas turbine blades and vanes formed of nickel-base, high temperature, corrosion-resistant alloys, provided with their protective coatings exhibit a greatly extended life at the same high temperatures and operating stresses, as compared with the identical blades or vanes which have not been subjected to the heat treatment of the present invention, while none of the desirable properties of such coated, and un-heat-treated parts are sacrificed.

The process of the present invention is particularly applicable to those nickel-base superalloys which are similar to those commonly referred to as "SM-200" and which have approximately the following specification analysis:

Although the actual analysis of SM-200 alloys supplied according to this specification analysis may vary somewhat beyond the ranges noted, the typical range of actual analyses of alloys to be treated according to the process of the present invention is as follows, expressed as weight percent:

	Percent
Chromium	8 to 10
Cobalt	9 to 11
Tungsten	11.5 to 13.5

2

Aluminum	4.75 to 5.25
Titanium	1.75 to 2.25
Boron	0.01 to 0.02
Zirconium	0.03 to 0.08

(but not more than 5 times boron content)

Carbon	0.12 to 0.17
Columbium	0.75 to 1.25
Nickel	Balance

	Impurities	Percent maximum
Manganese	0.2	
Sulfur	0.015	
Silicon	0.2	
Iron	1.5	
Copper	0.1	

The preferred analysis of such a nickel-base superalloy to be used in the process of the present invention consists of the following essential elements, in approximately the weight percentages set forth, although normal manufacturing deviations from this analysis may be tolerated in accordance with normal procedures in the production of nickel-base superalloys.

	Percent
Chromium	9.0
Cobalt	10.0
Tungsten	12.5
Columbium	1.0
Aluminum	5.0
Titanium	2.0
Iron	max
Boron	0.015
Carbon	0.15
Other elements as specified above.	
Nickel, balance, except for minor impurities.	

According to the process of the present invention, a gas turbine blade or vane cast from the nickel base alloy is preferably provided with a protective coating in accordance with the prior patent to Joseph, No. 3,102,044 of 1963, specifically in accordance with Examples 1, 2, 3, or 4 of said Joseph patent, but preferably at 2000° F. for a period of about four hours.

Any of these treatments provides the blade or vane member with a surface layer of a composition selected from the group of metals comprising aluminum, magnesium, chromium, columbium, cobalt, titanium, tantalum, tungsten, silicon, alloys thereof, oxides thereof and mixtures of the foregoing, which has been sintered on the surface of the blade or vane, and preferably comprises about 64% titanium, and 36% aluminum, the weight of the coating being about 30% of the weight of the coated vane or blade; or alternatively the coating consists of a mixture of finely divided particles of aluminum and silicon comprising about 90% aluminum and about 10% silicon.

According to the process of the present invention, the coated blades or vanes are subjected after coating to heat treatment and the preferred procedure for this coating treatment comprises heating the coated blade or vane members in vacuum or an inert gas, such as argon, or less preferably in air at 2000° F. for a period of about four hours, followed by about one to four hours heating at 2250° F., followed by heat treatment at 1600° F. for a period of from 32 to 64 hours; the step of normalizing by cooling in a gas, preferably an inert atmosphere, being carried out between each of the three heat treating operations. The heat treatment may be varied, while achieving the results of the present invention by initially heating the coated blade or vane at about 2000° F. for about 4 hours, normalizing by cooling in air or an inert at-

mosphere, heating at a temperature of from 2200° to 2260° F. for from one to 4 hours, followed by cooling in air or an inert gas, the heating at 1550° to 1650° F. for a period of from one to three days, again followed by air cooling or in an inert atmosphere.

Alternatively the heat treatment may comprise initially heating the coated vane or blade at an increasing temperature in the range of 1800° to 2200° or 2260° F., the temperature being increased at the rate of about 100° F. per hour, and thereafter holding the blade or vane at a temperature of 2200° to 2260° F., preferably 2250° F. for from one to 4 hours, all preferably in an inert atmosphere.

The cooling in gas is preferably in an inert atmosphere, such as argon, or less preferably in air.

Most preferably, the blades and vanes treated according to the present invention are not only made from an alloy of the specified composition, but are also characterized by the elongated, columnar grain structure having grain boundaries substantially parallel to the principal axis and with substantially no grain boundaries normal to the principal axis, all in accordance with the disclosure of the copending application of Francis L. Ver Snyder, Ser. No. 361,323, filed Apr. 17, 1964, now Patent No. 3,260,505 which described methods of producing such substantially uni-directionally oriented crystals forming either a blade or a vane member.

In the following table are shown test results on four different uni-directionally cast test specimens of the same composition, from the same heat, and which are differentiated, as follows:

Specimen A: Alloy SM-200, as cast.

Specimen B: Alloy SM-200, coated with a protective coating in accordance with the procedure set forth in the Joseph patent, involving heating at 2000° F. for four hours.

Specimen C: Alloy SM-200 un-coated, but heated for four hours in vacuum at 2000° F.

Specimen D: Alloy SM-200, coated the same as specimen B, but then heat treated after coating by heating in vacuum at 2250° F. for four hours, air cooling, and again heating for 64 hours at 1600° F., followed by air cooling.

When subjected to stress rupture tests at elevated temperatures (1400° F.) with a stress loading of 95,000 pounds per square inch, the following test results were obtained:

Specimen	Temp., °F.	Stress, p.s.i.	Life (hrs.)	Percent Elonga-	Percent Reduction in area
A-----	1,400	95,000	675.1	12.4	16.9
B-----	1,400	95,000	543.0	12.9	19.4
C-----	1,400	95,000	567.2	11.7	15.4
D-----	1,400	95,000	2,205.5+	13.9	17.4

Thus, the heat treatment of the test specimen increased the life of the stress rupture test specimen more than three times over life of any of the other specimens of the same alloy which had not been so heat treated.

The properties were obtained with no loss in oxidation resistance with respect to specimen B and are shown in the drawing where the oxidation resistance of material similar to specimens A, B and D are compared by measurement of weight gain with time in air at 2200° F. It is considered that there is no significant difference between the oxidation resistance of specimens B and D and therefore the heat-treatment described results in no loss in oxidation resistance but with improved properties in other respects.

It will be noted that specimen D, treated according to the present invention oxidized much less in air at elevated temperatures than the same alloy as cast (specimen A) and much the same as the coated but un-heat-treated specimen B, showing that the heat treatment at

2250° F. did not detrimentally affect the oxidation resistance imparted by the coating.

In the drawing, time in hours is plotted against increase in mass (weight) for a given area, expressed as milligrams weight gain for each one-hundredth of a square centimeter of alloy area is plotted against time.

Furthermore, no loss in high-temperature strength is observed as evidenced by the following data:

Specimen	Temp., °F.	Stress, p.s.i.	Life (hrs.)	Percent Elonga-	Percent Reduction in area
A-----	1,800	20,000	657.4	22.7	37.5
D-----	1,800	20,000	690.6	23.6	47.1

The invention in its broader aspects is not limited to the specific steps, processes and compositions shown and described but departures may be made therefrom within the scope of the accompanying claims without departing from the principles of the invention and without sacrificing its chief advantages.

What is claimed is:

1. The method of heat treating a nickel-base alloy to improve its stress rupture life at temperatures in excess of 1200° F. which comprises subjecting a nickel-base alloy having essentially the following composition, excluding incidental impurities:

	Percent
Chromium -----	8 to 10
Cobalt -----	9 to 11
Tungsten -----	11.5 to 13.5
Aluminum -----	4.7 to 5.25
Titanium -----	1.75 to 2.25
Columbium -----	0.75 to 1.25
Boron -----	0.01 to 0.02
Carbon -----	0.12 to 0.17
Zirconium -----	0.03 to 0.08

(but not more than 5 times the boron content), and the balance of the alloy consisting of nickel, and heat-treating such alloy by heating the alloy for a period of one to four hours at a temperature of about 2000° F., cooling the alloy in gas, heating the alloy to a temperature of from 2200° F. to 2260° F. for several hours, cooling the alloy in gas, reheating the alloy to a temperature in the range of from 1550° to 1650° F. for a period of one to three days, and again cooling the alloy in gas.

2. The method according to claim 1 in which the alloy is initially heated at 2000° F., is next heated at 2250° F., and is finally heated at 1600° F.

3. The method according to claim 1 in which the alloy has the following composition, excluding incidental impurities:

	Percent
Chromium -----	9
Cobalt -----	10
Tungsten -----	12.5
Columbium -----	1
Aluminum -----	5
Titanium -----	2
Boron -----	0.015
Carbon -----	0.15
Nickel -----	Balance

4. The method according to claim 2 in which the alloy has the following composition, excluding incidental impurities:

	Percent
Chromium -----	9
Cobalt -----	10
Tungsten -----	12.5
Columbium -----	1
Aluminum -----	5
Titanium -----	2

	Percent
Boron	0.015
Carbon	0.15
Nickel	Balance

5. The method of heat treating a nickel-base alloy to improve its stress rupture life at temperatures in excess of 1200° F. which comprises subjecting a nickel-base alloy having essentially the following composition, excluding incidental impurities:

	Percent
Chromium	8 to 10
Cobalt	9 to 11
Tungsten	11.5 to 13.5
Aluminum	4.75 to 5.25
Titanium	1.75 to 2.25
Columbium	0.75 to 1.25
Boron	0.01 to 0.02
Carbon	0.12 to 0.17
Zirconium	0.03 to 0.08

(but not more than 5 times the boron content), and the

balance of the alloy consisting of nickel, and heat-treating such alloy by heating the alloy for a period of one to four hours at a temperature of from 1800° to 2260° F., and holding the alloy at a temperature of from 2200° F. to 2260° F. for several hours, cooling the alloy in gas, reheating the alloy to a temperature in the range of from 1500° to 1650° F. for a period of one to three days, and again cooling the alloy in gas.

5 to 15

balance of the alloy consisting of nickel, and heat-treating such alloy by heating the alloy for a period of one to four hours at a temperature of from 1800° to 2260° F., and holding the alloy at a temperature of from 2200° F. to 2260° F. for several hours, cooling the alloy in gas, reheating the alloy to a temperature in the range of from 1500° to 1650° F. for a period of one to three days, and again cooling the alloy in gas.

15

balance of the alloy consisting of nickel, and heat-treating such alloy by heating the alloy for a period of one to four hours at a temperature of from 1800° to 2260° F., and holding the alloy at a temperature of from 2200° F. to 2260° F. for several hours, cooling the alloy in gas, reheating the alloy to a temperature in the range of from 1500° to 1650° F. for a period of one to three days, and again cooling the alloy in gas.

15 to 20

balance of the alloy consisting of nickel, and heat-treating such alloy by heating the alloy for a period of one to four hours at a temperature of from 1800° to 2260° F., and holding the alloy at a temperature of from 2200° F. to 2260° F. for several hours, cooling the alloy in gas, reheating the alloy to a temperature in the range of from 1500° to 1650° F. for a period of one to three days, and again cooling the alloy in gas.

20

#### References Cited by the Examiner

##### UNITED STATES PATENTS

2,887,420	5/1959	Llewelyn et al.	148-13.1
3,166,412	1/1965	Bieber	75-171
3,212,886	10/1965	Freedman	75-171
3,254,994	6/1966	Quigg	75-171
3,272,666	9/1966	Symonds	148-13

DAVID L. RECK, Primary Examiner.

R. O. DEAN, Assistant Examiner.