

[54] HEAT TREATMENT OF NICKEL ALLOYS
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[52] U.S. Cl..... 148/142, 148/31, 148/32.5, 148/162

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[51] Int. Cl..... C22c 41/02, C22f 1/10, C22c 39/20

Primary Examiner—C. Lovell

[58] Field of Search 148/12.7, 142, 162, 32.5, 148/124; 75/128 R, 128 G, 128 T, 171, 134 F

[57] ABSTRACT

[56] References Cited

Heat treating process enables obtaining desired combinations of strength, ductility and fabricability characteristics in heat resistant age-hardenable alloys having precipitation-hardening amounts of columbium, titanium and/or tantalum in a nickel-containing matrix.

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8 Claims, No Drawings

HEAT TREATMENT OF NICKEL ALLOYS

The present invention relates to age-hardenable nickel alloys and more particularly to heat treatment of nickel alloys, including nickel-iron-chromium alloys strengthened with columbium and titanium.

Age hardenable alloys based on nickel and/or iron and containing precipitation hardening amounts of titanium and/or columbium, and possibly with aluminum or other precipitation hardening elements, have been known and used for many years. Often, the alloys are strengthened with heat treatments comprising annealing or solution treating at high temperatures such as 1,700°F. or 2,100°F. or higher, cooling rapidly down from the solution temperature to room temperature, e.g., air cooling or water quenching, and thereafter reheating at lower temperatures of around 1,100°F. to 1,400°F. to precipitation harden the alloys. Precipitation-strengthened alloys containing chromium are often used for components of gas turbines, e.g., turbine blades and turbine rotor discs. For instance, one of the relatively recent additions to the family of nickel-iron-chromium age-hardenable alloys is described in U.S. Pat. No. 3,663,213.

While very substantial progress has been accomplished is providing high-strength age-hardened alloy articles, needs for improvements are continually arising. Greater strength, particularly including stress-rupture strength and yield strength, are frequently desired and special characteristics, e.g., resistance to low cycle fatigue, have become of increased importance. Ductility requirements are practically always present and experience with known alloys often brings forth needs for special improvements, e.g., notch ductility or capability for specially required elongation or reduction of area characteristics at a special temperature. Among other special needs are weldability, maintenance of required shape and size without distortion, resistance to high cycle fatigue, e.g., rotating beam load, corrosion resistance (including oxidation resistance), impact strength and stability after long time exposure in service. Improvements in the processing of known alloys are particularly desired for obtaining specially needed combinations of such important metallurgical characteristics and for facilitating production of desired articles and structures with use of presently available alloys. And, of course, process improvements in the present can become highly beneficial for enhancing the characteristics of future alloys.

In the present invention there has been discovered a process for heat treatment of age-hardenable nickel-containing alloys to provide articles having desired combinations of strength, fabricability and ductility, or other desired characteristics, in the heat treated condition.

It is an object of the present invention to provide a process for heat treating age-hardenable nickel alloys.

Other objects and advantages of the invention will become apparent from the following description.

The present invention contemplates a process for heat treating an age-hardenable heat-resistant nickel alloy containing precipitable amounts of gamma-prime forming metal selected from the group consisting of columbium, titanium and tantalum comprising: heating the alloy at a temperature in the gamma-prime solid solution range of the alloy and obtaining the alloy in the solid solution condition having precipitable amounts of

the gamma-prime forming metal in solid solution; cooling the alloy down to the gamma-prime solvus temperature; then slowly cooling the alloy uniformly at a controlled cooling rate not greater than 500°F. per hour, e.g., about 20°F. per hour to 500°F. per hour, from the gamma-prime solvus temperature down to at least about 100°F. below the gamma-prime solvus temperature, thus into precipitation temperature range of the alloy; heat treating the slow-cooled alloy at an upper temperature in the precipitation range to precipitate coarse particles of gamma-prime at (in the vicinity of) the grain boundaries of the alloy and dispersed uniformly within the grains while retaining a portion of the gamma-prime forming metal in solution; thereafter heat treating the alloy at a lower temperature in the precipitation hardening range to precipitate fine particles of gamma-prime dispersed uniformly within the grains; and then cooling the alloy to room temperature, thereby providing a precipitation hardened alloy having coarse particles of gamma-prime at the grain boundaries and having coarse gamma-prime particles and fine gamma-prime particles dispersed uniformly within the grains. The process may also precipitate the delta and eta equilibrium phases (Ni_3Cb and Ni_3Ti) in the grain boundaries.

When carrying the invention into practice, the slow cooling rate should be controlled sufficiently to avoid having the alloy at high elevated temperature for excessively long periods of time that would result in detrimental grain growth or excessive overaging. And, of course, production economy negates extending the heat-treatment time beyond benefit. Accordingly, for most purposes the slow-cooling rate should be at least about 20°F. per hour (°F/Hr) and is advantageously at least 50°F/Hr.

The age-hardenable heat-resistant nickel alloys treated in the process of the invention comprise, by weight, at least 2% metal from the group consisting of columbium, titanium and one-half the wt. % of any tantalum, at least about 25% nickel, up to 60% iron with a total of at least 50% nickel-plus-iron, and are characterized by a solidus temperature of at least about 2,300°F. Nickel is required for providing, inter alia, stability to the microstructure, including a stable austenite matrix; if the alloy does not contain sufficient nickel, detrimental phases, e.g., sigma, may be formed. Substantial amounts of chromium, e.g., 8% or advantageously 12% or more for corrosion resistance, can be present in the alloy. Small amounts of aluminum, e.g., 0.3% or 3% aluminum, may be present and can be beneficial for strength, ductility and/or oxidation characteristics. Thus, the process includes heat treatment of age-hardenable nickel-iron-chromium alloys, e.g., heat resistant alloys containing about 40% nickel, 40% iron, 15% chromium, 3% columbium, 1.7% titanium and 0.3% aluminum.

The gamma-prime of the particles precipitated in the heat treatment is the $\text{Ni}_3(\text{Cb},\text{Ta},\text{Ti})$ gamma-prime precipitate, which may also comprise other elements such as aluminum, e.g., $\text{Ni}_3(\text{Cb},\text{Ti},\text{Al})$. The solution temperatures are sufficiently high for enabling precipitable amounts of columbium, tantalum and/or titanium to enter into solid solution in practical solution treating times, e.g. ½ hour or 8 hours. Some columbium or titanium or other elements may be retained, possibly as carbides, without solution. Most of the solution temperatures are in a range of about 1,600°F. to 1,950°F.

Advantageously for high temperature strength, the solution treatment is at 1,625°F. to 1,700°F. when the columbium-plus-titanium-plus-½ tantalum content is 4% to 5.5% and is at 1,700°F. to 1,800°F. when the columbium-plus-titanium-plus-½ tantalum content is 5.7% to 6.7%, and the time is sufficient to obtain a homogenous gamma phase solution, e.g., one-half hour or more. Herein, percentage summations of tantalum plus other gamma-prime forming elements, the weight percentage of tantalum present is multiplied by one-half (in view of the relatively high atomic weight of tantalum). The precipitation-hardening temperature range spans the temperature at which, for most commercial practices, strengthening precipitates of the gamma prime can be precipitated in the alloy, e.g., 4 hours to 48 hours at 1,100°F. to 1,800°F. Advantageously, the coarse particles are precipitated in the upper one-third of the range and the fine particles are precipitated in the lower half of the range. The upper precipitation may be accomplished, and the coarse particles precipitated, by slow cooling through the precipitation range, provided that sufficient dissolved gamma-prime is retained for subsequently precipitating the fine particles.

Generally, the present process precipitates essentially all of the dissolved columbium, titanium and tantalum, with at least about 20% (by volume) of the gamma-prime in the coarse particle form and at least about 20% of the gamma-prime in the fine particle form. Inasmuch as essentially all of the gamma prime is precipitated when the process is complete, the process provides advantages of microstructural stability. Forms of the gamma-prime particles include plate-like, globular, and cubic shapes. The coarse particle sizes can be from about 0.04 to 1 micron and the fine particle sizes can be up to 0.1 micron, depending upon alloy composition. In the same alloy, the coarse particles are at least twice, usually five or ten times, the size of the fine particles. With a nickel-chromium-iron alloy composition containing not more than 2.5% aluminum-plus-titanium and at least 2.5% columbium, good results were obtained by precipitating coarse particles of 0.04 to 0.1 micron size and fine particles of sizes up to 0.02 microns.

Good results have been obtained with special embodiments wherein, in accordance with the invention, the alloy is cooled slowly from the solid solution temperature down through the precipitation range and thereafter reheated for one or more treatments in the precipitation range to complete the gamma-prime precipitation. For instance, nickel-iron-chromium alloys containing 4% to 5.3% columbium-plus-titanium are cooled slowly, at rates in the range of 50°F/Hr to 500°F/Hr, from solid solution temperatures in the range of 1,625°F. to 1,950°F., down to 1,100°F. or lower and thereafter reheated at least once in the range of 1,100°F. to 1,625°F. to finish precipitation.

An important feature of the invention is the provision of special embodiments whereby special benefits are achieved with heat treatments according to advantageously restricted ranges. Thus, advantageously long stress-rupture life in combination with good short-time tensile strength and ductility and good fabricability for welding or brazing are achieved with a triple-stage heat treatment of an age-hardenable nickel-iron-chromium alloy containing titanium, columbium, and aluminum according to a triple-stage treatment comprising: heating to a solid solution condition at least about 1,750°F.

or higher; slow-cooling at a rate of about 50°F/Hr to 500°F/Hr from the solid solution temperature to below the precipitation hardening range, e.g., slow-cooling to 1,100°F., and then cooling to room temperature at any desired rate, e.g., air cooling; reheating at an intermediate temperature of about 1,450°F. to about 1,625°F. for about 1 Hr. to 24 Hrs. and cooling to room temperature at any desired rate; and reheating to a precipitation temperature of about 1,275°F. to 1,425°F. and holding within this range for about 1 to 24 Hrs., cooling at a controlled rate of about 20°F/Hr to 200°F/Hr to a lower precipitation range of 1,100°F. to 1,200°F. and holding in this lower range for a total aging time of about 5 Hrs. to 24 Hrs. and thereafter cooling to room temperature at any desired rate.

Another embodiment, which is referred to herein as a two-stage treatment, achieves good stress-rupture life and tensile strength and advantageously high ductility and also has advantages of production economy and fabricability with heat treatment of an age-hardenable nickel-iron-chromium alloy containing titanium, columbium and aluminum comprising: heating to a solid solution condition at a temperature of about 1,675°F. or higher; slow-cooling at a rate of 250°F/Hr to 350°F/Hr from the solid solution temperature down through the precipitation-hardening range, e.g., down to 1,100°F., and then down to room temperature at any desired rate; and reheating to a precipitation temperature of about 1,275°F. to 1,425°F. and holding within this range for about 1 to 24 Hrs., cooling at a controlled rate of about 20°F/Hr to 200°F/Hr to a lower precipitation range of about 1,100°F. to 1,200°F. and holding in this lower range for a total aging time of about 5 Hrs. to 25 Hrs. and thereafter cooling to room temperature at any desired rate.

The foregoing two-stage and three-stage treatments referred to in connection with nickel-iron-chromium alloys containing titanium, columbium and aluminum particularly applicable in the heat treatment of age-hardenable nickel-iron alloys containing (in weight percentages) about 39% to 44% nickel, 14.5% to 17.5% chromium, 1.5% to 2% titanium, 2.5% to 3.3% columbium, 0.05% to 0.4% aluminum, up to 0.06% carbon, up to 0.35% manganese, up to 0.35% silicon, up to 0.006% boron and balance essentially iron. For treating alloys in this range, and possibly other alloys, heating three hours at 1,550°F. is recommended for the intermediate stage in three-stage embodiments of the invention and, also, a procedure of heating eight hours at 1,325°F., furnace cooling at 100°F/Hr to 1,150°F. and holding eight hours at 1,150°F. is recommended for the precipitation stage in two-stage or three-stage embodiments of the invention.

It is contemplated that the heat treatment of the invention is generally applicable for improving the metallurgical characteristics, or obtaining at least acceptable strength and ductility characteristics while avoiding detrimental effects of more rapid cooling from solution temperature, e.g., embrittlement, cracking, warping or other structural distortion, in processing of age-hardenable nickel alloys containing at least about 25% nickel, up to 60% iron, with a total of at least 50% nickel-plus-iron, up to 6.5% columbium and up to 5% titanium, up to 6% tantalum, with a total of at least 2% columbium-plus-titanium-plus-½-tantalum, up to 6.5% aluminum, provided the total of columbium, titanium, ½ tantalum and aluminum does not exceed 10%, up to

2% vanadium, up to 25% chromium, advantageously 12% to 25% chromium, up to 30% cobalt, up to 10% molybdenum or tungsten or mixtures thereof and up to 0.2% each of boron, zirconium and carbon. Either the nickel, or the iron when present, may be considered as the balance.

For the purpose of giving those skilled in the art a better understanding of the practice and advantages of the invention, the following illustrative examples are given.

EXAMPLE I

A nickel-iron alloy that had been hot rolled to nine-sixteenths inch diameter bar was obtained in the hot-rolled condition. Analyzed chemical composition of the alloy (Alloy 1) was 41.92% nickel, 16.28% chromium, 2.96% columbium, 1.90% titanium, 0.33% aluminum, 0.03% carbon, 0.003% boron, 0.14% manganese, 0.04% silicon, 0.01% copper, 0.001% sulfur and balance iron. (Percentage amounts of columbium referred to herein may include small incidental amounts of tantalum.) A specimen (Specimen I) of the bar of alloy 1 in the hot-rolled condition was heated to the solid-solution condition by heating one hour at 1,800°F. and was then slowly cooled directly from the 1,800°F. solid-solution temperature down to 1,100°F. at a slow cooling rate of 280°F/Hr; then the specimen was air cooled to room temperature. Next, in the present example of a two-stage embodiment of the invention, specimen I was precipitation heat treated at 1,325°F. for 8 hours, furnace cooled at a rate of 100°F/Hr to 1,150°F., for 8 Hrs. and then air cooled to room temperature. Metallurgical examination of the thus heat-treated specimen I showed the heat treatment had precipitated gamma-prime as coarse particles of about 0.04 to 0.1 micron size and as fine particles of 0.02 or less micron size dispersed uniformly in the grains and did not produce excessive amounts of needle phases. Tensile test bars and combination smooth-bar/notched-bar stress-rupture test bars were machined from the bar in the two-stage heat-treated condition and were tested at room temperature and at 1,200°F. by standard procedures. Results of 0.2% offset yield strength (YS) in kips per square inch (ksi), ultimate tensile strength (UTS), tensile elongation as percent along 1.0 inch gage length (E1) and reduction of area across .252 inch

of 0.178 inch, a gage length of 0.715 inch and a notch radius of .005 inch ($K_T=4.0$).

EXAMPLE II

Four other specimens (II-1, II-2, II-3 and II-4) of hot rolled nine-sixteenths inch diameter bars of the same alloy composition treated in Example I were solution treated at 1,800°F. for 1 Hr. and slowly cooled from the solution temperature to 1,100°F. at the following cooling rates: II-1 at 50°F./Hr, II-2 at 100°F/Hr; II-3 at 200°F/Hr and II-4 at 500°F/Hr; all were air cooled from 1,100°F. to room temperature. Thereafter, specimens II-1, II-2, II-3 and II-4 were reheated to an intermediate treatment temperature of 1,550°F. for 3 Hrs. and air cooled to room temperature and were then precipitation heat-treated by heating at 1,325°F. for 8 Hrs., furnace cooling at 100°F/Hr down to 1,150°F. and holding at 1,150°F. for 8 Hrs., which was followed by air cooling to room temperature. All four of the triple-stage heat-treated specimens had intra-granular dispersions of coarse particles and fine particles of gamma-prime. Results of short-time tensile tests and stress-rupture tests conducted by standard practices (in accord with Example I) on the heat-treated alloy products resulting from the four triple-stage embodiments in the present Example are set forth in Table I.

The heat treated conditions produced in Examples I and II were all characterized by good machinability, as confirmed by machining of threaded test bars.

Also shown in Table I are the results of comparable testing of the same alloy composition when treated by two different heat treatments, A and B, that are contrary to the invention. Two other specimens, A and B, of the nine-sixteenth-inch diameter hot rolled bar stock of alloy 1 were heat treated by solution treating one hour at 1,800°F. and air cooling to room temperature (which caused an average cooling rate of about 22,250°F. per hour between 1,800°F. and 1,100°F). Then, in treatment A the air-cooled alloy was further treated according to the precipitation heat treatment that followed the slow cooling of Specimen I in Example I, and in treatment B the air-cooled alloy was further treated according to the intermediate and precipitation treatments that followed the slow cooling of the specimens in Example II.

TABLE I

Treatment	Cool Rate	Age*	Short-Time Tensile Tests								Stress-Rupture Tests			
			Room Temperature				Smooth Bar 1200°F.				Combination Smooth/Notch Bar 105 Ksi at 1200°F.			
			YS, ksi	UTS, ksi	E1, %	RA, %	YS, ksi	UTS, ksi	E1, %	RA, %	Life Hr.	E1, %	RA, %	Fracture Location
I	280°F/Hr	1	159.0	195.5	20	39	121.0	141.0	31	60	94.4	14	31	Smooth Section
II-1	50°F/Hr	2	147.0	185.5	17	24	126.0	144.0	22	37	138.3	18	37	do.
II-2	100°F/Hr	2	150.5	186.0	17	26	129.0	145.0	24	42	136.8	18	38	do.
II-3	200°F/Hr	2	150.5	190.5	18	24	128.5	147.0	24	43	138.5	18	44	do.
II-4	500°F/Hr	2	146.0	185.0	18	25	125.0	143.5	28	50	131.8	22	40	do.
A	Air Cool	1	159.0	185.5	25	49	123.0	146.0	30	55	1.8	—	—	Notch Section
B	Air Cool	2	154.0	186.0	19	31	123.0	141.0	30	56	77.5	18	48	Smooth Section

*Age (1)—1325°F. for 8 Hrs., Furnace Cool at 100°F/Hr to 1150°F. and hold for 8 Hrs. at 1150°F., then Air Cool to room temperature.

*Age (2)—1550°F. for 3 Hrs., Air Cool, plus 1325°F. for 8 Hrs., Furnace Cool at 100°F/Hr to 1150°F and hold for 8 Hrs. at 1150°F, then Air Cool to room temperature.

diameter gate section (RA) and stress-rupture test results of life in hours, elongation and reduction of area are set forth in the following Table I. Stress-rupture test results were obtained with a smooth section diameter

Referring to the results in Table I, it is observed that the product of treatment I displayed high reduction of area in short-time tensile testing, which is considered characteristic of advantageously good low-cycle fatigue

characteristics. Also, this example wherein the rate of cooling from solid solution was controlled within a range of 250°F/Hr to 350°F/Hr provides a good combination of tensile strength, stress-rupture strength and ductility and offers the production economy of the shorter two-stage treatment. Turning to treatment II, it is evident that superior stress-rupture life along with desirable levels of tensile strength and ductility was obtained with the three-stage treatment, especially with cooling rates of 100°F/Hr to 200°F/Hr.

Treatment A failed to provide satisfactory notch ductility inasmuch as the stress-rupture test specimen fractured in the notched section after a very short life at 1200°F.

While the invention has been exemplified herein with the specific composition of alloy 1, it is contemplated that the heat treatment be performed with many other alloys having compositions within ranges herein provided and be beneficial for obtaining improved metallurgical characteristics, particularly including advantageously good ductility, and also enhanced stress-rupture life and other desirable characteristics mentioned hereinbefore. For instance, the invention is considered applicable in heat treatment of alloys 2 to 21 having the nominal compositions set forth, below the nominal composition of alloy 1, in the following Table II.

TABLE II

Alloy	Nominal Compositions, Weight Percent														
	C	Mn	Si	Cr	Co	Mo	W	Cb	Mg	Ti	Al	B	Zr	Fe	Ni
1	0.03	0.1	0.1	16	—	—	—	3.0	—	1.9	0.3	0.003	—	Bal.	42
2	0.05	0.1	0.1	12	—	5.7	—	—	—	2.8	0.2	0.015	—	Bal.	42
3	0.05	0.3	0.2	15	—	4.0	4.0	—	—	3.0	1.0	0.010	—	27.0	Bal.
4	0.05	0.2	0.3	22	—	9.0	—	4.0	—	0.2	0.2	—	—	3.0	Bal.
5	0.12	0.1	0.3	15	29	3.7	—	—	—	2.2	3.0	—	—	0.7	Bal.
6	0.04	0.2	0.3	19	—	3.1	—	5.0	—	0.9	0.4	—	—	18.5	Bal.
7	0.04	0.6	0.2	15	—	—	—	—	—	2.4	0.6	—	—	6.5	Bal.
8	0.06	—	—	15	—	3.0	3.0	3.0	.02	0.6	0.4	0.005	0.03	7.0	Bal.
9	0.15	0.5	0.5	20	10	10.0	—	—	—	2.6	1.0	0.005	—	—	Bal.
10	0.06	0.1	0.7	20	1	—	—	—	—	2.5	1.3	—	—	—	Bal.
11	0.07	0.5	0.7	20	18	—	—	—	—	2.4	1.4	—	—	—	Bal.
12	0.09	—	—	19	11	10.0	—	—	—	3.1	1.5	0.005	—	—	Bal.
13	0.02	—	—	5	—	—	—	—	—	2.5	0.6	0.005	—	Bal.	42
14	0.03	—	—	—	15	—	—	3.0	—	1.4	0.6	0.008	—	Bal.	38
15	0.08	—	—	18	18	4.0	—	—	—	2.9	2.9	0.006	0.05	—	Bal.
16	0.05	—	—	19	12	6.0	1.0	—	—	3.0	2.0	0.005	—	—	Bal.
17	0.08	—	—	15	18	5.2	—	—	—	3.5	4.3	0.030	—	—	Bal.
18	0.08	—	—	19	13	4.3	—	—	—	3.0	1.3	0.006	0.006	—	Bal.
19	0.06	—	—	15	15	5.3	—	—	—	3.5	4.4	0.03	—	—	57
20	0.10	—	—	20	20	—	—	—	—	3.0	2.0	—	—	—	Bal.
21	0.04	0.70	0.3	15	—	—	—	0.9	—	2.5	0.8	—	—	6.8	Bal.

The present invention is particularly applicable in the production of nickel-iron alloy products and articles for use where strength and ductility are required in structures, including welded structures, engines and other machines, and in articles, including components of machines, e.g., gas turbine blades, and is specially applicable for overcoming distortion difficulties that may otherwise arise when heat treating large or complex structures and articles, e.g., turbine rotor discs. Among other things, the invention is useful in the production of turbine shafts and cases, diffuser cases, compressor discs and shafts and fasteners.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are consid-

ered to be within the purview and scope of the invention and appended claims.

We claim:

1. A process of heat treating an age-hardenable heat-resistant nickel alloy consisting essentially of at least about 25% nickel and up to 60% iron, with a total of at least 50% nickel-plus-iron, and precipitable amounts of gamma-prime forming metal selected from the group consisting of up to 6.5% columbium, up to 5% titanium and up to 6% tantalum and mixtures thereof with a total of at least 2% columbium-plus-titanium-plus-½ tantalum up to 6.5% aluminum, the total of the columbium content plus the titanium content plus the aluminum content plus one-half the tantalum content does not exceed 10%, up to 2% vanadium, up to 25% chromium, up to 30% cobalt, up to 10% molybdenum, tungsten or mixtures thereof, up to 0.2% boron, up to 0.2% zirconium and up to 0.2% carbon and characterized by a solidus temperature of at least about 2,300°F., a gamma-prime solvus temperature of at least 1600°F. and a gamma-prime precipitation temperature of at least 1100° F. comprising:

a. heating the alloy at a temperature of about 1,600 to 1,950°F and gamma-prime solid solution range of the alloy and obtaining the alloy in the solid solution condition having precipitable amounts of the

gamma-prime forming metal in solid solution; b. cooling the alloy from the solid solution temperature down to the gamma-prime solvus temperature; c. then slowly cooling the alloy uniformly at a controlled cooling rate of at least about 20°F. per hour and not greater than 500°F. per hour from the gamma-prime solvus temperature down to about 1,100°F or lower; d. heat treating the slow-cooled alloy at an upper temperature in the precipitation range to precipitate coarse patches of gamma-prime in the vicinity of the grain boundaries of the alloy and dispersed uniformly within the grains while retaining a portion of the gamma-prime forming metal in solution; e. thereafter heat treating the alloy at a lower temperature in the precipitation hardening range to precipitate fine particles of gamma-prime dispersed uniformly within the grains; and

f. then cooling the alloy to room temperature, to thereby provide the alloy in the heat treated condition characterized by having coarse gamma-prime particles at least twice the size of the fine gamma-prime particles.

2. A process as set forth in claim 1 wherein the alloy is slowly cooled from the solid-solution temperature down through the precipitation hardening temperature range at a slow-cooling rate in the range of 50°F. per hour to 500°F. per hour.

3. A process as set forth in claim 2 wherein the slow-cooling rate is in the range of 100°F. per hour to 200°F. per hour.

4. A process as set forth in claim 2 wherein the slow-cooling rate is in the range of 250°F. per hour to 350°F. per hour.

5. A process as set forth in claim 1 wherein, after the alloy is slow-cooled to at least 100°F. below the gamma-prime solvus temperature, the reheating at an upper temperature is at about 1,450°F. to about 1,625°F. for about 1 to 24 hours and the reheating at a lower temperature is accomplished by reheating at about 1,275°F. to 1,425°F. for about 1 to 24 hours, then cooling at a rate of about 20°F. per hour to 200°F.

per hour to a range of about 1,100°F. to 1,200°F. and holding at about 1,100°F. to 1,200°F. for at least 5 hours.

6. A process as set forth in claim 1 wherein, the solid solution temperature is at least about 1,675°F., the alloy is slow cooled from the solid solution temperature down through the precipitation hardening temperature at a rate in the range of 250°F. per hour to 350°F. per hour and is thereafter further heat treated by reheating at about 1,275°F. to 1,425°F. for about 1 to 24 hours, then cooling at a rate of about 20°F. per hour to 200°F. per hour to a range of about 1,100°F. to 1,200°F. and holding at about 1,100°F. for at least 5 hours.

7. A process as set forth in claim 1 wherein the alloy contains about 39% to 44% nickel, 14.5% to 17.5% chromium, 1.5% to 2% titanium, 2.5% to 3.3% columbium, 0.05% to 0.4% aluminum, up to 0.06% carbon, up to 0.35% manganese, up to 0.35% silicon, up to 0.006% boron and balance essentially iron.

8. A process as set forth in claim 7 wherein the coarse particles are precipitated with sizes of 0.04 microns to 0.1 microns and the fine particles are precipitated with sizes of up to 0.02 microns.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 3,871,928

DATED : March 18, 1975

INVENTOR(S) : Darrell Franklin Smith, Jr. and Edward Frederick
Clatworthy

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 1, line 26, for "is" read --in--.

Col. 3, line 8, insert the word "in" before the word
"percentage".

Col. 4, line 38, insert the word "are" after the word
"aluminum".

Col. 6, line 11, for "," read --;--.

Col. 8, line 25 (line 21 of claim 1) insert "in the"
after the word "and".

Line 57 (line 35 of claim 1) for "or" read --and--.

Line 60 (line 38 of claim 1) for "patches" read
--particles--.

Col. 10, line 13 (line 10 of claim 6) after "1,100°F." insert
--to 1200°F.--.

Signed and Sealed this

twenty-sixth Day of August 1975

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks