METHOD OF FABRICATING AND HEAT-TREATING PRECIPITATION-HARDENABLE NICKEL-BASE ALLOYS

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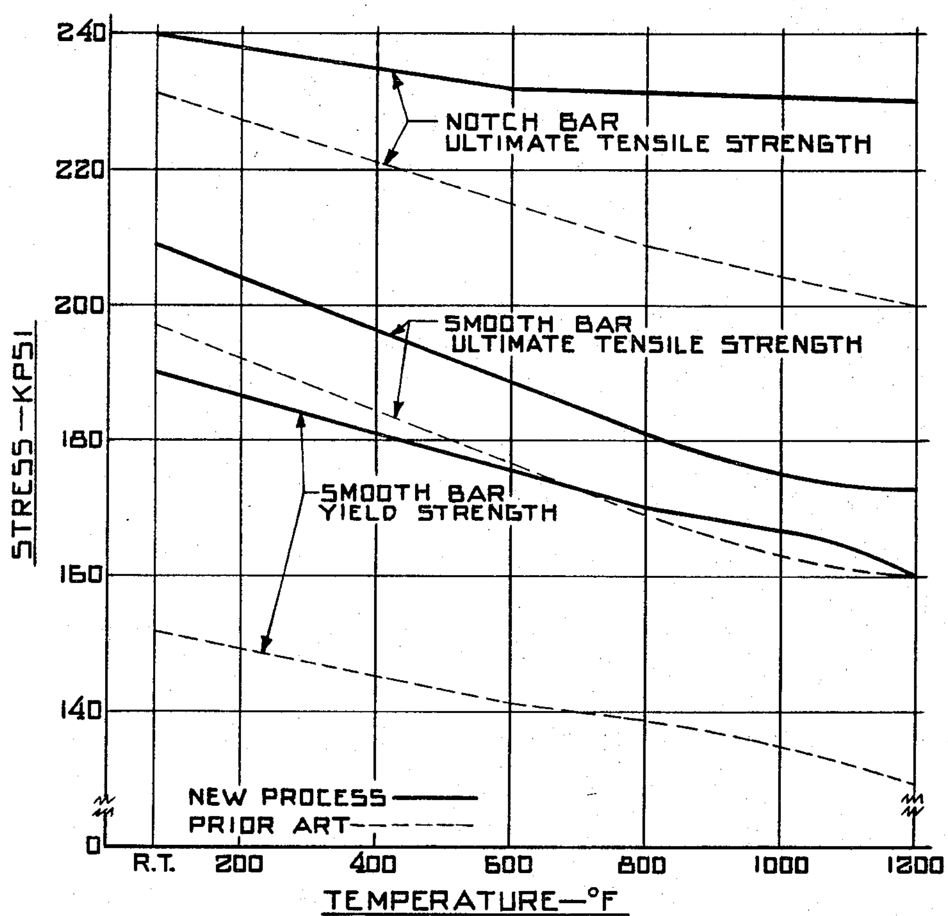

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1

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METHOD OF FABRICATING AND HEAT-TREATING PRECIPITATION-HARDENABLE NICKEL-BASE ALLOYS

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ABSTRACT OF THE DISCLOSURE

A method of fabricating and heat-treating certain precipitation-hardenable nickel-base alloys to improve strength and hardness without substantial diminution of ductility, by hot-working the material about a hundred degrees or more below its solutioning temperature, and then heat-treating the alloy at a temperature several hundred degrees below the solutioning temperature.

This invention relates to a method of forming and heat-treating alloys of the precipitation-hardenable or age-hardenable type, to yield products of higher hardness and strength than those of the prior art.

It has previously been known that the strength of precipitation-hardenable high-temperature materials can be altered or controlled within limits by changes in the composition of the alloy, by varying the grain size, or by changing the grain flow orientation. Accordingly, the prior art practices of fabrication and heat-treatment are primarily based on these three variables, and the procedures employed are those expected to give the best compromise for the desired effect in a given situation.

In the present invention it has been found that control of a fourth variable, namely, material dislocations, during fabrication and heat-treatment would result in additional strength and hardness, as well as other desirable properties.

It is known that in the atomic lattice structures of metals there are many imperfections, known as material dislocations. If the atomic lattice of each crystal in any given piece of metal were perfect, its strength would be far higher than any known today, owing to the enormous force required to distort or rupture the lattice. However, since material dislocations exist, owing to imperfect positioning of atoms or to the presence of more or less atoms in a lattice than are required for a perfect structure or to the rejection of atoms by precipitation from the lattice, there are formed slip planes along which it is relatively easy for movement to take place. This results in metals having only the strengths commonly known in the art.

It is also known that cold-working of metals increases the number of material dislocations, and that such increase does not result in weakening or softening the metal, but on the contrary hardens and strengthens it. This is brought about by the fact that when the number of dislocations exceeds some characteristic value for a given metal, they tend to interlock and inhibit the slip planes from being operative.

Cold-working, however, is an expensive process, owing to the low formability of the metal in the cold state, the high tool-pressures and power required, and short tool life. Further, it leaves the metal in a state of locked-in internal stress which may result in distortion, cracking, or embrittlement. For this reason, when a part has undergone any substantial cold-working treatment, it is usual to anneal or stress-relieve it by heat, which of course removes also the hardness and strength achieved by cold-working.

2

It is an object of this invention to increase the number of material dislocations in a metal without significantly increasing internal stresses.

It is another object to provide a method of hot-working of metals below the solution-annealing temperature, followed by heat-treatment at a still lower temperature to promote formation and precipitation of a secondary phase.

A further object is to provide an improved method of hot-working and heat-treating precipitation-hardenable alloys.

The foregoing objects and other ancillary thereto will be readily understood on reading the following specification in connection with the accompanying drawing, in which there is shown a graph comparing the tensile strength at various temperatures of material processed according to the invention, with the strength of material processed according to the prior art.

The precipitation-hardenable alloys are normally forged or otherwise hot-worked, in the prior art, in a temperature range approximating the solutioning temperature of the alloy, and are subsequently annealed or heat-treated for aging at a temperture below solutioning temperature but approaching it. There may be several successive aging treatments at progressively lower temperatures. In such prior art processing, the rate of regression at the solutioning temperature is so high that any dislocations introduced by the hot-working immediately regress to the solution state and do not persist in the metal. At the high temperatures of subsequent heat-treatment, precipitation of carbides or inermetallic compounds does occur, but there is still a relatively high rate of regression, so that a certain proportion of such precipitates goes back into solution. Finally, the prolonged and successive heat-treatments at high temperatures results in considerable grain growth.

In the present invention it has been found that significantly higher strength and hardness, and smaller grain size, without important loss of ductility, may be achieved in precipitation-hardenable alloys by starting forging or other hot-working procedures definitely below the solutioning temperature, continuing working as the metal cools to a temperature well below the solutioning temperature, and following by a single heat-treatment generally of the order of several hundred degrees below the aging temperatures previously employed, to promote the formation and precipitation of a secondary phase. The hot-working temperatures range from about two hundred to several hundred degrees below the solutioning temperature, and the subsequent aging heat-treatment may be as much as a thousand degrees or more below the solutioning temperature.

These procedures result in a lower regression rate and avoid the growth of the large grains inherent in the processes of the prior art, and in effect approximate a simultaneous cold-working and stress-relieving treatment, whereby the increase in material dislocations of cold-working is retained without the locked-in internal strains of true cold-working as previously known.

One of the precipitation-hardenable nickel-base alloys is sold under the trademark Udimet 700, and has the following nominal composition.

	Percent
Carbon	0.1
Chromium	15
Cobalt	19
Molybdenum	5
Titanium	3.5
Aluminum	4.5
Iron, max.	4
Boron	0.03
Nickel	Balance

The prior art procedure for hot-working Udimet 700 is to begin work at 2150° F. and not to continue it below 1800° F., reheating if the work is not finished by the time it cools to that temperature. This is followed by four successive heat-treatments, 2150° F. for 4 hours followed by air cooling, again reheat to 1550° F. for 24 hours with air cooling, and then heat to 1400° F. for 16 hours and again followed by air cooling.

EXAMPLE I

Udimet 700 was processed according to the invention by hot-working in the range from 1900° F. down to 1700° F., followed by a single aging heat-treatment of approximately 1400° F. for 6 hours, followed by air cooling down to room temperature. This new procedure results in tremendous saving of time over the four heat-treatments of the prior art. Table I shows the improvement in properties resulting, as compared with Udimet 700 processed according to the prior art.

TABLE I

	Testing temperature	Ultimate tensile strength (p.s.i.)	.2% offset yield strength (p.s.i.)	Elongation, percent	Reduction of area, percent
Prior art proc.	Room temp.	200,000	140,000	29	30
	1,200° F.	185,000	124,000	26	28
New proc.	Room temp.	228,000	174,000	21	28
	1,200° F.	207,000	154,000	18	18

Udimet 700 processed according to the invention therefore has an ultimate tensile strength 12–14% greater than the prior art material, and a yield strength 22–24% greater. These strength improvements also are accompanied by improvement in hardness, and in the creep, fatique, and stress rupture properties of the metal.

Another useful nickel-base precipitation-hardenable alloy is that known as Inconel 718, having the nominal composition given below.

	Percent
Carbon	0.03–0.10
Chromium	17.0–21.0
Cobalt, max.	1.00
Molybdenum	2.80–3.30
Titanium	0.65–1.15
Aluminum	0.40–0.80
Boron, max.	0.006
Nickel	50.0–55.0
Manganese, max.	0.35
Silicon, max.	0.35
Phosphorus, max.	0.015
Sulfur, max.	0.030
Copper, max.	0.10
Columbium	5.00–5.50
Iron	Balance

The elements specified as maxima are associated with some of the components as common impurities, for which the further refining necessary to eliminate them is impractical or unnecessary. Commercial columbium containing a minor amount of tantalum is acceptable in this formulation.

The prior art procedure for Inconel 718 is hot-working at 1900° F., with a subsequent heat-treatment at 1750° F. followed by air cooling, reheat to 1325° F. and hold for 8 hours, then furnace cool to 1150° F. and hold for 10 hours, and finally air cooling to room temperature.

EXAMPLE II

Inconel 718 was processed according to the invention by hot-working at approximately 1800° F., then given a single aging heat-treatment at 1325° F. for 8 hours followed by furnace cooling to 1150° F. and held at 1150° F. for a total aging cycle of 18 hours. Thus the hot-working was not only at a substantially lower temperature, but the first high-temperature heat-treatment of the prior art was eliminated. Table 2A shows the improvement in properties resulting, as compared to Inconel 718 processed according to the prior art.

TABLE 2A

	Testing temperature	Ultimate tensile strength (p.s.i.)	.2% offset yield strength (p.s.i.)	Elongation, percent	Reduction of area, percent
Prior Art Proc.	Room temp.	197,000	152,000	19	36
	800° F.	169,000	139,000	20	46
	1,000° F.	163,000	135,000	18	41
	1,200° F.	160,000	130,000	19	44
New Proc.	Room temp.	209,000	185,000	20	36
	800° F.	181,000	170,000	20	37
	1,000° F.	175,000	167,000	18	38
	1,200° F.	173,000	160,000	19	44

It will be seen that Inconel 718 processed according to the invention had an ultimate tensile strength 6–8% greater at the various testing temperatures than the prior art material, and a yield strength 22–24% greater, without loss of ductility. Hardness tests of material hot-worked and heat-treated according to the prior art showed values of 21 R_c in the as-forged condition and 43 R_c in the heat-treated condition. Hardness tests of new-processed material showed 37 R_c in the as-forged condition and 46 R_c heat-treated. Tests of creep strength, fatigue strength, and stress rupture strength also showed significant improvements. As an example of such improved properties, Table 2B shows the results of fatigue testing of smooth samples by the R. R. Moore procedure of prior art Inconel 718, and Inconel 718 processed according to the invention. All samples were tested at 800° F.

TABLE 2B

	Stress applied (p.s.i.)	Number of cycles	Result
Prior art proc.	75,000	134,000	Failed.
	70,000	188,000	Do.
	65,900	304,000	Do.
	62,500	331,000	Do.
	60,000	10,000,000	Runout.
New proc.	100,000	391,000	Failed.
	95,000	1,720,000	Do.
	90,000	7,795,000	Do.
	87,500	3,052,000	Do.
	85,000	1,076,000	Do.
	80,000	12,000,000	Runout.
	70,000	102,000,000	Do.

The enormous improvement in fatigue strength of material processed according to the invention is immediately apparent from Table 2B. The new process sample fatigue-tested at 100,000 p.s.i. survived a greater number of cycles than any of the prior art samples tested above 60,000 p.s.i. At stresses from 80,000 p.s.i. up to 95,000 p.s.i. the new process samples survived a very much larger number of cycles before failure than any of the prior art samples tested from 62,500 p.s.i. up. The fatigue endurance strength for prior art material is about 60,000 p.s.i., whereas for the inventive material it is about 80,000 p.s.i. or a little higher.

Udimet 630 is another nickel-base precipitation-hardenable alloy, having the following nominal composition.

	Percent
Carbon, max.	0.04
Chromium	15.0–19.0
Cobalt, max.	1.0
Molybdenum	2.7–3.4
Titanium	0.7–1.5
Aluminum	0.2–1.0
Boron	0.001–0.007
Manganese, max.	0.2
Silicon, max.	0.2
Phosphorum, max.	0.015
Sulfur, max.	0.015
Columbium	5.8–6.8
Iron	16.0–19.0
Tungsten	2.7–3.4
Nickel	Balance

Here again, commercial columbian containing a small proportion of tantalum is acceptable.

The prior art procedure for processing Udimet 630 is hot-working at 1900° F., followed by an annealing treatment at 1850° F. for 2 hours followed by air cooling to room temperature, then an aging heat-treatment of 1400° F. for 10 hours, furnace cooling to 1200° F., and holding at that temperature for 10 hours.

EXAMPLE III

According to the invention, Udimet 630 was hot-worked at approximately 1700° F., followed by an aging heat-treatment at 1400° F. for 10 hours, furnace cooled to 1200° F., and held for 10 hours. This processing avoids the high temperature of forging of the prior art, and eliminates the previous high temperature anneal. Table 3 shows some of the improved properties resulting, compared with properties achieved by the prior art method.

TABLE 3

	Testing temperature	Ultimate tensile strength (p.s.i.)	.2% offset yield strength (p.s.i.)	Elongation, percent	Reduction of area, percent
Prior Art Proc.	Room temp.	212,000	181,000	5	7
	1,200° F.	215,000	181,000	9	7
New Proc.	Room temp.	232,000	212,000	13	24
	1,200° F.	231,000	213,000	12	16

From Example III it will be seen that the processing Udimet 630 according to the invention resulted in an increase in ultimate tensile strength of about 7%, and an increase in yield strength of about 18%, at both testing temperatures. Further, there was a considerable increase in ductility at both testing temperatures. Creep strength, fatigue strength, and stress rupture properties were also improved.

The drawing shows, by way of example of the improved precipitation-hardenable materials, a graphic comparison of some of the tensile properties of Inconel 718, the solid lines being the plotting for material processed according to the invention, and the dashed lines for material processed according to the prior art. The notch bar tensile tests show that the inventive treatment does not render the material notch sensitive, the values for the new material remaining consistently higher than those for prior art material. For smooth bars, ultimate tensile strength and yield strength at .2% offset are also higher at all temperatures for the new material. Of particular interest is the yield strength, since this parameter is of greater design significance than ultimate strength. It will be noted that the values for material produced by the new process are much higher than the yield strengths for the previously known material, and are in fact in the region of ultimate strength for the prior art.

Although the invention has been described above in a preferred embodiment, it will be understood by those skilled in the art that various modifications may be made without departing from the inventive principle. The temperatures for hot-workng and for aging treatment will vary in accordance with the particular metal being processed, but will still follow the principle of hot-working below the solution-annealing temperature, followed by heat-treatment to promote formation and precipitation of a secondary phase. It is intended to cover all such variations in the appended claims.

What is claimed is:

1. A method of fabricating and heat-treating precipitation-hardenable nickel-base alloys selected from the group consisting of Udimet 700, Inconel 718, and Udimet 630, comprising hot-working said alloy at a temperature at least one hundred degrees below the solutioning temperature of said alloy to induce strain hardening and material dislocations, and then heat-treating said alloy at a temperature at least four hundred degrees below said solutioning temperature to promote formation and precipitation of a secondary phase.

2. The method of fabricating and heat-treating precipitation-hardenable nickel-base alloys as recited in claim **1**, wherein said hot-working temperature is not over 1900° F.

3. The method of fabricating and heat-treating precipitation-hardenable nickel-base alloys as recited in claim **1**, wherein said selected alloy is Udimet 700, said hot-working temperature is between 1900° F. and 1700° F., and said heat-treating temperature is approximately 1400° F. for approximately 6 hours, followed by air cooling.

4. The method of fabricating and heat-treating precipitation-hardenable nickel-base alloys as recited in claim **1**, wherein said selected alloy is Inconel 718, said hot-working temperature is approximately 1800° F., and said heat-treating temperature is approximately 1325° F. for approximately 8 hours, followed by furnace cooling to 1150° F. and held at 1150° F. for a total aging cycle of approximately 18 hours.

5. The method of fabricating and heat-treating precipitation-hardenable nickel-base alloys as recited in claim **1**, wherein said selected alloy is Udimet 630, said hot-working temperature is approximately 1700° F., and said heat-treating temperature is approximately 1400° F. for approximately 10 hours, followed by furnace cooling to 1200° F. and held at 1200° F. for approximately 10 hours.

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