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THERMOMECHANICAL PROCESSING OF INTERMEDIATE SERVICE TEMPERATURE NICKEL-BASE SUPERALLOYS

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8 Claims

ABSTRACT OF THE DISCLOSURE

The nickel-base superalloys, such as Inconel 718 and Incoloy 901, are strengthened by thermomechanical processing, usually including solutioning, cold work, precipitation aging and warm working. The processing results in a fine-grained microstructure containing dispersed intermetallic particles and a dense dislocation substructure which may be stabilized by post-deformation precipitation aging.

BACKGROUND OF THE INVENTION

The present invention relates in general to the nickel-base alloy field and, more particularly, to the thermomechanical processing of the intermediate service temperature nickel-base superalloys for improved strength.

In a copending application of Owczarski et al. entitled, "Thermomechanical Strengthening of the Superalloys," Ser. No. 864,268, filed Sept. 26, 1969, there is disclosed a combination of thermal and deformational heat treatments to strengthen the high γ' volume fraction nickel-base superalloys. That application is, in turn, a continuation-in-part of prior application, Ser. No. 746,013, filed July 19, 1968 and now abandoned. The present application is directed to the processing of the nickel-base superalloys characterized by a lower γ' volume fraction and, in addition, subject to eta phase precipitation.

In another copending application of the same assignee entitled, "Processing of Nickel-Base Alloys for Improved Fatigue Properties," E. E. Brown et al., Ser. No. 38,227 filed May 18, 1970 the fatigue properties of the lower γ' volume fraction nickel-base superalloys, such as Inconel 718 and Incoloy 901, are improved by a thermomechanical processing sequence involving, in these particular alloys, generation of a spheroidal eta pinning phase with subsequent recrystallization to provide a fine-grained microstructure. The process of the instant application also provides a fine-grained microstructure, typically on the order of ASTM 11-14, and in addition provides the capability of additional strengthening.

Unlike the high γ' volume fraction superalloys, such as Udimet 700, which have a stable γ' strengthening precipitate, both Inconel 718 and Incoloy 901 are strengthened by metastable coherent γ' -type precipitates which are formed by aging in the 1100°-1500° F. temperature range. In Incoloy 901, a face-centered-cubic Ni_3Ti γ' phase forms, while in Inconel 718 both face-centered-cubic $\text{Ni}_3(\text{Al}, \text{Ti})$ γ' and body-centered-tetragonal Ni_3Cb γ'' are present. Moreover, in both of the latter alloys a stable intermetallic phase forms at higher temperatures (1400°-1800° F.) at the expense of the ordered strengthening γ' and/or γ'' phases. Udimet 700 has no analogous intermetallic phase. These stable phases are a hexagonal Ni_3Ti (901) and orthorhombic Ni_3Cb (718) and, as these alloys are conventionally processed, both are generally considered to be detrimental to the strength and toughness of the alloy if present in substantial quantities.

Representative chemistries of the alloys herein discussed are as follows:

	Inconel 718	Incoloy 901	Udimet 700
Ni.....	(1)	(1)	(1)
Cr.....	18.5	12.5	15
Co.....	0.1	0.1	0.07
C.....	18	34	18.5
Fe.....	0.9	-----	3.3
Ti.....	0.6	2.6	4.3
Al.....	3	6	5
Mo.....	5	0.015	0.03
Others.....	(Cb+Ta)	(B)	(B)

¹ Balance.**SUMMARY OF THE INVENTION**

This invention contemplates the duplex processing of certain precipitation-hardened nickel-base superalloys for strength improvements. It is applicable to the intermediate service temperature nickel-base superalloys of the type characterized by Inconel 718 and Incoloy 901.

In the processing sequence, the alloys are first thermomechanically processed to refine the morphology of the intermetallic phase, utilizing it for a grain boundary pinning function, and are subsequently thermomechanically processed to introduce and stabilize dislocation arrays within the microstructure.

The general steps in the duplex processing are:

a solution anneal plus rapid cooling;
cold working;
a high temperature age;
and warm working.

Usually low temperature aging to precipitate the γ' and γ'' phases is also conducted.

A particularly preferred processing of the Incoloy 901 alloy comprises:

heat treatment at about 2000° F. for 2 hours with water quench;
cold working to reductions in area of 75 percent;
precipitation aging at 1700° F. for about 16 hours with fast air cool;
and warm working at about 1725° F.

For the Inconel 718 alloy the following processing is preferred:

heat treatment at 1950° F. for 2 hours with fast air cool;
cold working to reductions in area of 75 percent;
precipitation aging at 1650° F. for about 16 hours with fast air cool;
and warm working about 1700° F.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The application of thermomechanical processing for substantial strengthening of the lower γ' volume fraction nickel-base superalloys has been occasionally attempted. In previously reported efforts (Thermomechanical Treatments Boost Alloy Strengths, J. C. Uy, Metals/Materials Today, Mar. 14, 1967) these alloys were cold deformed in a partially or completely hardened condition. The alloys were difficult to work, required specialized deformation techniques and, while some increases in yield strengths were shown, ductility was severely limited.

In the present process, the alloys are solution annealed and cooled sufficiently rapidly from the solution temperature to suppress the formation of the strengthening precipitate phase. The material is then cold-worked to introduce a dislocation substructure, the deformation generally varying over a wide range (15-75%). In fact, in the absence of the strengthening precipitate, the material can be cold worked to reductions in excess of 75 percent reduction-in-area (1.39 true strain) without difficulty. At this time the dislocation substructure may be stabilized

by normal precipitation aging which is most effective when there is an interaction between the dislocations and the formation of the precipitating phase.

In the preferred duplex processing, however, a high temperature age is utilized, subsequent to the cold working sequence, to precipitate the eta phases. The cold worked substructure provides intragranular nucleation sites which lead to a favorable morphology and distribution of precipitate when subsequently heat treated above the γ' solvus for eta phase precipitation. The alloys are then warm worked at a temperature where recrystallization can take place, utilizing the precipitate to establish a fine grain size. Generally, deformations within the range of 15-75 percent (about .2-1.4 true strain) are utilized.

The warm work operation provides compatibility with subsequent processing wherein the dislocation substructure is stabilized by normal precipitation aging. In this case, following warm working in the grain refinement sequence, fast cooling is required to preserve the dislocation substructure during the cooling sequence. Also the degree of deformation is preferably restricted to the 15-75 percent deformation band.

Two as-received bars of Incoloy 901 (1" diameter) and Inconel 718 (1½" diameter) were utilized. Deformation was carried out by swaging with reductions of 6 percent per pass. During high temperature swaging, the material was given a ten minute reheat between passes.

The following table illustrates the representative processing provided:

TABLE I

	901	718
Solution.....	2,000° F./2 hrs./WQ	1,950° F./2 hrs./F.A.C.
Cold work.....	75% R.A. (1.39E)	75% R.A. (1.39E)
Precipitation age.....	1,700° F./16 hrs./F.A.C.	1,650° F./16 hrs./F.A.C.
Warm work.....	50% at 1,725° F., fast cool.	50% at 1,700° F., fast cool.
Aging for.....	1,000-1,400° F./16 hrs. 1,325° F./8 hrs. 1,200° F./16 hrs./A.C.	1,000-1,400° F./16 hrs.

Hardness and fatigue measurement indicate that these alloys are strengthened if cold worked prior to precipitation aging. The property improvements observed are attributed primarily to the stabilization of the dislocation arrays in the substantially worked alloys by their interaction with the coherent strengthening precipitates. Two types of interactions are possible:

(1) In Incoloy 901, the γ Ni₃Ti interacts with dislocations to form stacking faults within the precipitate particles. The dislocation substructure is stabilized since motion of a dislocation away from a particle would require a high energy defect within the γ' .

(2) In Inconel 718, the highly strained coherent precipitates appear to nucleate preferentially at dislocations such that the elastic strain field around the dislocation accommodates that of the precipitate. This type of dislocation-precipitate interaction stabilizes a dislocation substructure by making it energetically unfavorable for dislocations to move away from their low energy sites.

In the duplex processing, grain refinement plus strengthening is provided. The hardness and strength of these alloys so processed is substantially increased. The fine grain size, dispersion of intermetallic particles and dislocation substructure are all important factors in this increase.

The stability of these microstructures has been demonstrated at temperatures up to 1325° F. which is typical of temperatures actually encountered by these alloys in service. The dislocation substructure is stabilized at these temperatures by the interactions of the dislocations with the strengthening precipitates according to the mechanisms previously discussed. To determine the stability of these alloys at more elevated temperatures, samples of duplex-processed material (901) were aged for 1, 4 and 16 hours at 1700° F. Following exposure at 1700° F., the hardness values decreased from an initial value of R_c 23, 20.5 and

19, respectively. Electronmicroscopy of the sample aged for 4 hours revealed complete recovery of the dislocation substructure, while the eta phase distribution and grain size were retained. Thus, it may be seen that the warm worked substructure contributes substantial hardness to the alloy.

After aging to form the strengthening precipitates, the duplex-processed alloys exhibit higher hardnesses than those conventionally processed. This can be attributed to the stabilized warm worked dislocation substructure. In utilizing this strengthening effect care must be taken to cool sufficiently rapidly from the warm working temperature to limit recovery. However, in applications where only grain size is of concern, rapid cooling is not necessary.

Strength improvements attained by the duplex processing described herein are summarized in Table II.

TABLE II

Property	Material condition (Alloy 901)		
	Normal HT	Duplex processing	Solution, CW plus direct age
Room temp.:			
0.2% yield (K s.i.)	120	188	242
UTS (K s.i.)	170	232	246
Percent elongation	18	7.7	6
Percent R of A	25	7.4	16.4
1,100° F.:			
0.2% yield	114	169	196
UTS	140	183.7	199
Percent elongation	13	8.6	5
Percent R of A		10	24.6

Thermomechanical treatments thus appear to be a suitable and practical method for strengthening the low γ' volume fraction superalloys such as Inconel 718 and Incoloy 901. One such strengthening treatment is simply to solution cold work and age for the strengthening precipitate. A second thermomechanical treatment, duplex-processing, consists of solution, cold work, high temperature age and warm work. This results in a fine grained microstructure, ASTM 11-14, containing dispersed intermetallic particles (eta) along with a dense dislocation structure. This may be stabilized by aging for the strengthening γ' precipitate. While dislocation recovery occurs above the γ' solvus, the fine grain size is maintained at higher temperatures.

The thermomechanical treatments presented are convenient from a view point of suitability to engineering application. All working operations may realistically be incorporated into a rolling or forging process. Also, the eta phase precipitation sequence and warm working operation may be combined. Thus, while the invention has been described in detail in connection with several preferred embodiments and examples, no limitation is specifically intended thereby. The invention in its broader aspects is not limited to the exact details described, but improvements to and departures therefrom may be made within the scope of the appended claims without departing from the principles of the invention and without sacrifice of its chief advantages.

What is claimed is:

1. The method of strengthening a precipitation-hardening nickel-base alloy characterized by the presence of a thermodynamically metastable γ' -type precipitate which comprises:

solution annealing the alloy and rapidly cooling the same to suppress the formation of the γ' -type precipitate; cold working the alloy in compression to a true strain of at least about 0.2;

precipitation heat treating the alloy at a temperature above the γ' -type precipitate solvus temperature, and warm working the alloy in compression to a true strain rate of at least about 0.2 and effecting recrystallization.

2. The method according to claim 1 wherein: the alloy is subjected to post deformation aging to precipitate the γ' -type precipitate in a homogeneous distribution.

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3. The method of strengthening the Incoloy 901 alloy which comprises:
 solution annealing the alloy at about 2000° F. and water quenching the same;
 cold working the alloy to a true strain of about 0.2–1.4; 5
 precipitation aging the alloy at about 1700° F. and fast cooling the same; and
 warm working the alloy at about 1725° F. and rapidly cooling the same.
4. The method according to claim 3 wherein: 10
 the alloy is subjected to post deformation aging at a temperature of 1000°–1400° F.
5. The method according to claim 4 wherein:
 the alloy is aged at about 1300°–1350° F. for a minimum of about 6 hours; 15
 and subsequently aged at about 1175°–1225° F. for a minimum of about 12 hours.
6. The method of strengthening the Inconel 718 alloy which comprises:
 solution annealing the alloy at about 1950° F. and 20
 rapidly cooling the same;
 cold working the alloy in compression to a true strain of about 0.2–1.4;

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- precipitation aging the alloy at about 1650° F. and rapidly cooling the same; and
 warm working the alloy at about 1700° F. and rapidly cooling the same.
7. The method according to claim 6 wherein:
 the alloy is subjected to post deformation aging at a temperature of about 1000°–1400° F.
8. The method according to claim 7 wherein:
 the alloy is aged as follows:
 1300°–1350° F. for a minimum of about 6 hours; and
 1125°–1175° F., the total aging time being about 18 hours.

References Cited

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