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[54] THERMOMECHANICAL STRENGTHENING OF THE SUPERALLOYS

[72] Inventors: William A. Owczarski, Cheshire; John M. Oblak, Rocky Hill, both of Conn.

[73] Assignee: United Aircraft Corporation, East Hart-

ford, Conn.

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 746,013, July 19, 1968, abandoned.

[52] U.S. Cl......148/12.7, 148/158, 148/162

[58] Field of Search............148/11.5, 12.3, 12.7, 12, 158, 148/142, 162

[56]

References Cited

UNITED STATES PATENTS 3,147,155 9/1964 Lamb......148/11.5

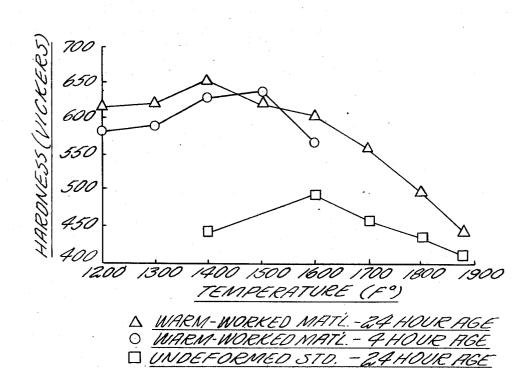
Primary Examiner—Richard O. Dean Attorney—Richard N. James

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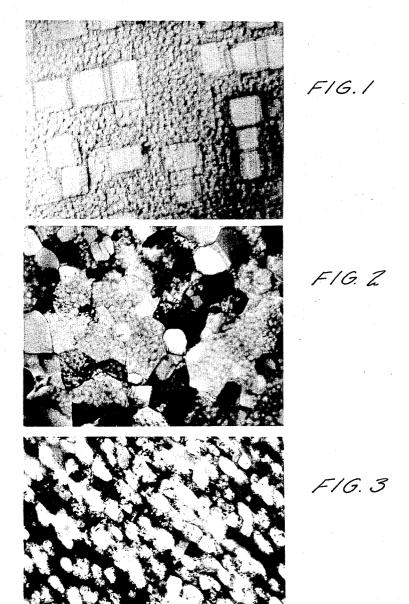
ABSTRACT

The superalloys are strengthened in a process involving both thermal and deformational treatments under controlled conditions. The method is particularly effective for the nickel-base superalloys of the γ - γ' -type having a volume fraction of the γ' phase exceeding about 25 percent at room temperature, and for the superalloys precipitating the topologically closepacked phases such as the sigma phase. It relies on the establishment of a microstructure wherein the γ' phase is precipitated in a uniformly distributed array having an interparticle spacing not exceeding about 5 microns; warm working the alloy to effect an area reduction of at least 15 percent: and subjecting the alloy to a stabilization heat treatment. The strength increase is attributable to a particular thermally and mechanically stable array of microcrystalline imperfections thus established and, in those alloys precipitating the sigma phase, also by an altered sigma phase morphology.

16 Claims, 7 Drawing Figures



SHEET 1 OF 3



INVENTORS. WILLIAM A. OWCZARSKI JOHN M. OBJAK Picharl N. James.

SET 2 OF 3

F16.4

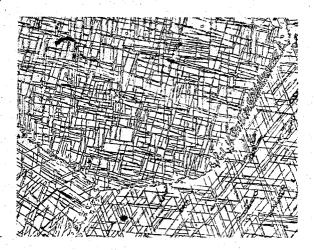
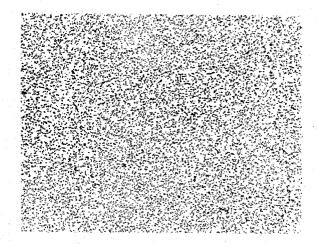


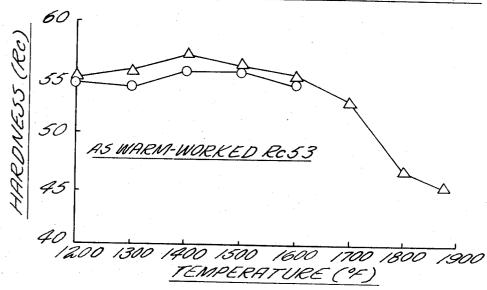
FIG.5



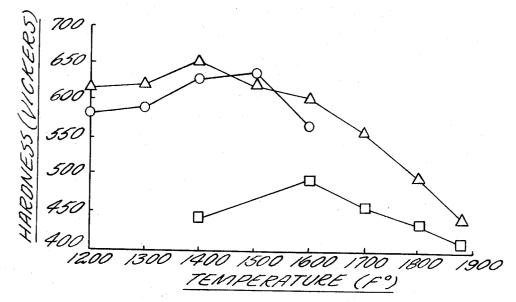
SHEET 3 OF 3

F16.6

D WARM-WORKED MAT'L-24 HOUR AGE ○ WARM-WORKED MAT'L- 4 HOUR AGE



F16.7



DEFORMED STD. - 24 HOUR AGE

□ UNDEFORMED STD. - 24 HOUR AGE

THERMOMECHANICAL STRENGTHENING OF THE **SUPERALLOYS**

This application is a continuation-in-part of application Ser. No. 746,013 filed July 19, 1968 and now abandoned.

BACKGROUND OF THE INVENTION

The present invention is most conveniently characterized as a metal processing technique and it is particularly adapted to improving the mechanical properties of the nickel-base and cobalt-base superalloys.

The superalloys are, in general, those alloys which display very high strengths at very high temperatures and, thus, which have significant utility in the fabrication of gas turbine engine components. The typical nickel-base superalloy of this type, 15 for example, is essentially a nickel-chromium solid solution (γ phase) hardened by the additions of elements such as aluminum and titanium to precipitate an intermetallic compound (γ') phase). The usual intermetallic compound, which is represented by the formula Ni₃(Al, Ti), is an ordered facecentered-cubic structure with aluminum and titanium at the corners of the unit cell and nickel at the face centers. These alloys also normally contain cobalt to raise the solvus temperature of the γ' phase, refractory metal additions for solution strengthening, and carbon, boron and zirconium to promote 25 topologically close-packed (TCP) phases which typically form ductility and fabricability. In the monocrystal form, these alloys may have reduced quantities of carbon to prevent crackproneness associated with the formulation of MC-type carbides.

ticular applicability are those nickel-base superalloys having a quantity of the γ' precipitate exceeding about 25 volume percent at the hot working temperatures and which is stable within the matrix at this same temperature. Representative of the alloys of this type are those identified in the industry as fol- 35

Designation	Composition (by weight)				
Udimet 700	15%Cr, 18%Co, 3.25%Ti,				
	4.25% Al, 5% Mo, 1%C,				
B-1900	0.03B, balance Ni 8%Cr, 10%Co, 1%Ti, 6% Al,				
MAR-M200	6% Mo, 0.11%C, 4.3%Ta, 0.07%Zr, 0.15%B balance Ni 9%Cr, 10%Co, 2%Ti, 5% Al, 12.5%W, 0.15%C, 1%Cb, .015%B, 0.05%Zr,				
	balance Ni				

Conventional alloys of the type described above find extensive usage in the gas turbine engine industry. As part of the continuing development programs, improvements in the properties of the superalloys are sought with or without significant alterations of the alloy chemistry. One very common problem with the typical superalloy, which usually represents a balance between strength and oxidation resistance among other criteria, relates to its inherent susceptibility to damage by such phenomena as high cycle and low cycle fatigue. Ac- $_{60}\,$ ing procedure: cordingly, a number of the development programs are directed toward the improvement of such specific properties without diminution of the other advantageous physical characteristics of the alloys in question. The thermomechanical strengthening sequence of the present invention is the out- 65 come of one such program.

In a copending application of the same assignee entitled Fabrication Method for the High Temperature Alloys, Ser. No. 692,705, filed Dec. 22, 1967, now U.S. Pat. No. 3,519,503 a simultaneously-applied elevated temperature- 70 compressive working operation was described to impart forgeability to the advanced gas turbine alloys. Similarly, in an other copending application of the same assignee entitled Method to Impart Fabricability to the Nickel-Base Superalloys, Ser. No. 745,958, filed July 19, 1968, now abandoned 75

the same problem with the superalloys, namely a lack of fabricability, was attacked by establishment of a particular polygonal subcell microstructure as attained by heat treatment and working. The present invention utilizes somewhat similar processing, particularly in the early sequences of the method, but ultimately achieves a thermally and mechanically stable array of microcrystalline imperfections for strengthening purposes.

Another problem often encountered with the superalloys is 10 the formation of undesirable phases during exposure to elevated temperatures or to some particular operating regime. The phase known as the sigma phase (σ) is a particularly well known example of a precipitate of this general type and many of the commercially available superalloys, such as INCO 901 and INCO 718, are specifically formulated to avoid the formation of these undesirable phases. Typically, the sigma forms as hard, brittle platelets which provide the natural sites for mechanical weakness and, in addition, compete with the γ matrix phase for the solid solution strengthening elements. A detailed discussion of the sigma phase may be found in an article by E. O. Hall et al. The Institute for Metals, Vol. 11 (1966) p. 61.

Sigma belongs to a class of intermetallic phases identified as in a platelike morphology in a size comparable to the alloy grain size. These TCP phases, such as the sigma, chi or mu phases, occur in both the nickel-base and cobalt-base alloys.

One series of alloys to which the present invention has par- 30 tion strengthened stainless steels may be changed from the detrimental platelet morphology to a less harmful globular shape by cold working. See, for example "The Effect of Sigma Phase on the Short-Time High Temperature Properties of 25 Chromium-20 Nickel Stainless Steel," Guarneiri et al., Trans. ASM, Vol. 42 (1950) p. 981. However, the use of cold work for this purpose in the nickel-base superalloy systems, for example, is generally not advisable. First, the cold-worked microstructure is inherently unstable in these superalloys at 40 their representative service temperatures. Secondly, the distribution of deformation in the cold-worked alloys and, consequently, the sigma phase configuration is apt to be quite heterogeneous. Furthermore, in the superalloy field cold work is normally difficult to achieve following full aging and 45 frequently introduces embrittlement or other microstructural instabilities if provided prior to aging.

SUMMARY OF THE INVENTION

The present invention provides a method for improving the yield and tensile strengths and improving the creep and fatigue resistance of the advanced nickel-base superalloys by thermomechanical means. The improved properties are provided by a stable array of microcrystalline imperfections established by controlled heat treatment and deformation, and preserved and stabilized by subsequent heat treatment causing further γ' precipitation and in the case of the alloys prone to sigma formation, a finely dispersed precipitate of equiaxed sigma.

Basically, the method disclosed herein involves the follow-

- 1. An initial microstructure is established by heat treatment involving solutioning plus aging to provide a microstructure consisting of a uniformly distributed γ' phase having an effective interparticle spacing not exceeding about 5 microns and a volume fraction not less than about 25 percent.
- 2. The material so heat treated is deformably processed at elevated temperatures subject to the following condi
 - a. the deformation temperature is selected to preserve the volume fraction and distribution previously established and usually corresponds to the aging temperature of step (1);
- b. the deformation temperature must not exceed either the solvus temperature of the γ' phase or the gross

recrystallization temperature and must not be less then the minimum recovery temperature;

- c. total deformation must be equivalent to at least about a 15 percent reduction (but is not likely to exceed a reduction of about 60 percent).
- 3. Following deformation the material is heat treated at a temperature which must not exceed the temperature of deformation and prior aging. This final heat treatment at lower temperatures is utilized to stabilize the microdefect array and additionally strengthen the alloy by normal 10 tion are particularly applicable. precipitation hardening. In this final heat treatment there is usually precipitation of additional γ' and, as appropriate, precipitation of the TCP phases. For certain alloys, a single heat treatment may suffice; for other alloys multiple heat treatments may be preferred, depending upon the optimum precipitation temperatures for the various phases and the properties desired in the finished product.

A significant measure of strengthening may be achieved in either of two microcrystalline forms, the generation of which in the alloys of the γ - γ' type is dependent upon the particular temperature within the aging temperature range at which the aging and deformation is accomplished. The more significant strengthening is achieved by working the alloy nearer the lower end of the aging temperature range, i.e., nearer the minimum recovery temperature. Processing at this temperature results in a warm-worked substructure comprising a randomly nonoriented, homogenous dislocation distribution.

When the material is deformed at a temperature nearer the 30upper end of the aging temperature range, i.e., nearer the solvus temperature of the γ' precipitate, a polygonal substructure is achieved and the dislocations are mobile enough to align

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Throughout the description reference is made to various alloys, temperatures, heat treatments and other process parameters. As utilized, the terminology employed herein is generally consistent with that utilized in the art. The following table sets forth certain select properties for several of the advanced superalloys which are of particular interest in the gas turbine engine industry and to which the methods of the present inven-

TABLE I

5	Alloy	Solidus, melting point	Lowest γ' solvus	Approximate recovery temperature 1	γ' aging temperature ²
:0	MAR-M200	2300	2250	1850	1850-2100
	B-1900	2300	2200	1850	1850-2050
	Udimet 700	2220	2100	1800	1800-1975

Coincident with the upper temperature limit for planar slip

It will be understood that the above temperatures are representative, certain of the above parameters being dependent to some extent on the exact alloy composition and prior history.

The representative conditions utilized to establish specific substructures in certain of the superalloys are set forth in Table II.

TABLE II.-NOMINAL CONDITIONS TO ESTABLISH SPECIFIC SUPERALLOY SUBSTRUCTURE (° F.)

		Pre-work heat treatment		Working		
Alloy	Substructure	Solution	Age	temperature range	heat treatment	
Udimet 700		A has	1,925-1,975/	1, 925-1, 975	Normal stabilize and final age.	
Udimet 700		2,100-2,150/	4 hrs. 1,800–1,850/	1, 800-1, 850	Do.	
MAR-M200		2,200-2,250/	4 hrs. 1,975–2,025/ 4 hrs.	1, 975-2, 025	Do.	
MAR-M200	Warm worked	2,200-2,250/ 4 hrs.	1,900-1,950/ 4 hrs.	1,900–1,950	Do.	

Note.—Times are those utilized for convenience and represent no limitation on the maximum or minimum allowable times. Aging and working at temperature levels between the bands listed above will likely produce mixtures of substructure types.

themselves at subcell boundaries to provide a regular array of defects along these boundaries.

In some cases, by processing the material at one tempera- 50 ture condition followed by subsequent processing at another condition, or by processing at intermediate temperatures, it is possible to produce both kinds of dislocation arrays in the microstructure

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a photomicrograph taken utilizing electron microscopy techniques showing the microstructure of a Udimet 700 alloy sample subjected to a conventional strengthening heat treatment. (Mag. 24,000 \times)

FIG. 2 is a photomicrograph of a Udimet 700 alloy sample processed to produce the polygonal substructure of the present invention. (24,000×)

FIG. 3 is a photomicrograph of the same alloy processed to produce the warm-worked substructure of the present invention. $(24,000\times)$

FIG. 4 is a photomicrograph of a sigma prone alloy processed conventionally. (500×)

FIG. 5 is a photomicrograph of the alloy of FIG. 4 processed according to the present invention. (500x) The sections shown in FIGS. 4 and 5 have been stain etched to reveal only the sigma and carbide phases.

FIOS. 6 and 7 are graphs comparing the hardness of the treatments prone alloys as conventionally processed and as processed according to the present invention.

In performing a solution heat treatment, the conditions are normally selected to dissolve the maximum quantity of the γ' precipitate into solid solution. In the case of materials like Udimet 700, all of the γ' phase can be solutioned. With MAR-M200 most, but perhaps not all, of the γ' phase is dissolved. Solution heat treatments are conducted near or above the solvus temperature for the particular alloy involved but 55 below the solidus temperature or that at which "hot shortness" occurs. In the case of the highly alloyed materials such as MAR-M200, where the solvus temperature approaches the solidus temperature, solutioning may actually be done slightly below the solvus temperature.

Aging of the alloy results in precipitation of the γ' phase. In the context of the present invention, aging may be undertaken at any temperature above the minimum recovery temperature, or that temperature above which nonplanar slip occurs, and below the solvus temperature of the γ' phase. Aging and working near the upper end of this range promotes formation of a polygonal substructure, while aging and working near the lower end of the range results in a warm-worked microcrystalline array. A further qualification on the aging parameters is established, particularly in a temperature-time relationship, to precipitate a minimum quantity of the γ' phase of about 25 volume percent at an interparticle spacing not exceeding about 5 microns.

Deformation is preferably undertaken at the same tempera ture at which the alloy has been aged. However, it may be undertaken at any elevated temperature within the aging range

² Preferred, in context

provided the microstructure established in aging is essentially maintained. The total deformation must exceed that equivalent to about a 15 percent reduction of area, and the strength increase which finally results is usually achieved in the range of a 15-60 percent reduction. This is not to say that 5 further reduction cannot be made without deleterious effect, but rather that the maximum advantages in physical properties will have been achieved at the point where a 60 percent reduction has been achieved.

Deformation is normally accomplished with a 5-10 percent 10 reduction per pass with reheat between passes to reestablish the temperature. With greater degrees of deformation, particularly when imposed in a limited number of passes, the synergistic effects of working plus external heat are necessarily considered It is desirable to prevent precipitation simultaneously with the working process, hence, the desirability of working the alloy at the same temperature at which it was aged is established. And, as previously mentioned, the selecfluenced by the particular form of strengthening desired, i.e., the polygonal or warm-worked microstructure or combination thereof.

The final postwork heat treatment comprises a normal stabilization and precipitation aging. The temperature in the 25 postwork heat treatment sequence must not exceed the temperature of deformation and final aging. Its purpose is to promote the final arrangement of the microcrystal imperfections into a stable array in the matrix through the further precipitation of the γ' phase and, in alloys containing more 30 than about 0.05 weight percent carbon, to precipitate intragranular carbides. Both of these subsequent precipitation events further stabilize the microdefect array and additionally strengthen by normal precipitation hardening.

A number of specific materials were tested under various 35 conditions. The following examples are representative of the preferred embodiments described herein. Further specific details relative to the present process will be evident to those skilled in the art, not only from the processing details set forth, but also from the evaluation of the various specimens so processed.

EXAMPLE 1

Polycrystalline Udimet 700 was thermomechanically processed to produce strengthening through achievement of a polygonal microstructure in the following manner:

Solution anneal at 2,140° F./4 hrs., air cool; age 1,925° F./4 hrs.; air cool; work at 1,925° F. to approximately a 60 percent reduction in area followed by stress relief at 1,925° F./2 hrs.; final age at 1,550° F./4 hrs. and 1,400° F./16 hrs.; and slow 50 cool to room temperature.

The physical property improvements were verified by various test procedures. These are set forth for Samples 1-5 in the following summary.

TENSILE PROPERTIES

Sample	temp s		0.2% yield Ultimate strength, K s.i. K s.i.		Percent R of A.	
1	80 80 80 1,000 1,200 1,200 1,400 1,400	202 210 140 189 182 124 142 120	265 267 200 244 226 180 166 150	11.7 9.6 17 6.7 18.3 16 29 33	11. 4 10. 6 20 9. 6 41 20 49	

Creep Properties

Thermomechanically treated samples tested at 100 k.s.i., 1,200° F., showed 0.1 percent creep in 72 hours. The normal creep for the untreated alloy is 0.1 percent creep in about 20

Stress Rupture Properties

Thermomechanically treated samples were tested at 125 k.s.i., 1,200° F. and exhibited failure in 790 hours. Comparable times for the untreated alloy average about 200 hours.

EXAMPLE 2

A monocrystal specimen formed from low carbon Udimet 700 was thermomechanically treated to produce a polygonal substructure as follows:

Solution anneal at 2,140° F./64 hrs.; age at 1,925° F.;4 hrs.; work at 1,950° F. to 41 percent area reduction; then slow cool to room temperature.

With this specimen the 0.2 percent yield strength was 152 k.s.i. For the untreated alloy the 0.2 percent yield strength is 121 k.s.i.

A similar treated specimen, tested in low cycle fatigue at 1,400° F. and 1.6 percent total strain amplitude, had a life of 3.093 cycles. The comparable lifetime of an untreated monocrystal of the same composition is about 900 cycles, that of an untreated polycrystalline sample of the same composition about 200 cycles.

EXAMPLE 3

A monocrystal sample of low carbon MAR-M200 was thertion of the aging—working temperatures is additionally in- 20 momechanically treated to produce a polygonal substructure as follows:

> Solution anneal at 2,250° F./100 hrs.; age at 2,000° F./2 hrs.; work at 2,000° F. to a 42 percent reduction followed by a slow cool to room temperature.

The 0.2 percent yield strength of this sample was 173 k.s.i. which compares to a 140 k.s.i. yield strength for the untreated alloy. In low cycle fatigue at room temperature with a 1.5 percent strain amplitude, life was 3.038 cycles as compared to a lifetime of about 700 cycles for the untreated monocrystal.

EXAMPLE 4

A Udimet 700 polycrystalline specimen was thermomechanically processed to produce a warm-worked substructure as follows:

Solution anneal at 2,140° F./4 hrs.; age at 1,825° F./4 hrs.; work at 1,800° F. to a 30 percent area reduction (specimen 1) and a 60 percent area reduction (specimen 2); then age as shown. Specimen

0	Specimen	Hardness after age at temperature shown (Rc)			
		1400° F.	1600° F.		
	1	46	45		
5	. 2	49	48		
	untreated but aged	40	38		

The physical property improvements verified by the foregoing data were achieved without change in alloy composition by the provision of a specific substructure by thermomechanical treatment of the alloy. The substructure size is of the order of 5 microns or less and was achieved because the original particle size and distribution of the γ' precipitate, as established by heat treatment, provided a network of particulate barriers with a spacing of 5 microns or less. The strength increase established is maintained as long as the substructure is maintained, and, hence, is evident up to at least the minimum recovery temperature for the particular alloy involved.

Either of the two types of dislocation structures previously mentioned while providing strengthening in and of themselves also provide means for significant strengthening in another fashion. When they are produced by working the superalloys at temperatures above the TCP phase precipitation range they inhibit the precipitation of these phases in the platelike Widmanstatten form, modifying the shape of the sigma, for example, to the more innocuous globular form and decreasing the actual precipitate particle size as clearly evidenced in FIGS. 4 and 5. The homogeneity of deformation prevents extensive nucleation of the sigma phase in the typical platelike mode and the stability of the dislocation structure prevents extensive recovery at the sigma aging temperature.

An alloy was designed to be highly sigma prone. Material was vacuum induction melted and cast with electrodes which were subsequently consumable arc melted under vacuum into

two 1 inch diameter bars. Chemical analyses of both bars were performed using atomic absorption (Cr, Ti, Mo, Co, Al) spectroscopy (B, Zr) and combustion (C) methods. The results are shown in Table III.

The bars were solution annealed at 2,150° F. for 4 hours and 5 fast air cooled. Bar A was then aged at 1,975° F. for 4 hours to precipitate the γ' phase. It was then swaged to a 60 percent reduction in area at 1,975° F. using a reduction of 6 percent per pass with a 10 minute reheat between passes. No difficulty was experienced during the working operation. Bar B was given an 1,875° F. age for 4 hours. Swaging at 1,875° F. resulted in severe cracking during the first swaging pass. The 1,875° F. age was found to promote the precipitation of both γ' and the platelike sigma.

TABLE III.—COMPOSITION BY WEIGHT PERCENT

	Cr	Ti	Мо	Co	Al	В	Zr	C	Ni
Bar A	16. 5 16. 5	4.6 4.6	$\substack{5.2\\5.2}$	$\begin{matrix} 16.5 \\ 16.3 \end{matrix}$	5.0 5.1	.005	<.001 <.001	. 025	Bal. Bal.

Bar A was sectioned and given final heat treatments of 4 and 24 hrs. at temperatures varying from 1,200° F. to 1,875° F. Specimens from this group, prepared according to the present invention, are hereinafter referred to as the warmworked specimens. In summary, these were processed as fol- 25

l. solution anneal	
2. age for	
3. swage	

2,150° F./4 hours 1,975° F./4 hours 60%RA at 1.975° F.

1,200°-1,875° F.; 4 or 24 hours

For the purposes of comparison, samples were prepared which had an identical thermal history but no deformation processing. Specimens of this type are hereinafter referred to 35 as standards.

The hardness of the warm-worked material was significantly higher than that of conventional nickel-base superalloys. As shown in FIG. 6, hardness values in the R_c 50's were obtained after aging at temperatures below 1,700° F. For comparison, 40 the hardness of fully heated treated Udimet 700 is - R. 39. In fact, the hardness of the sigma containing material is nearly equivalent to the low R_c 60 values of high-speed tool steel. For this alloy, a maximum hardness of R_c 57.5 was obtained after a 24 hour age at 1,400° F. The Vickess hardness curves of FIG. 45 7 indicate that the peak hardness after a 4 hour age is nearer 1,500° F. The shift in peak hardness to 1,400° F. with increasing aging time indicates that any softening mechanisms, such as recovery or coarsening, occurring at 1,400° F., are more then compensated, presumably by continued precipitation of 50 phase and, optionally, a topologically close-packed phase the sigma phase.

As in the case of hardness, the yield strength of the warmworked material in compression was significantly higher than that of the conventionally processed nickel-base superalloys, as shown in Table IV. At room temperature, the ductility of 55 the hardest warm-worked specimen is above that of tool steel but the yield strength is 100,000 p.s.i. lower. However, at 1,000° F. the strength of the tool steel has dropped significantly while the warm-worked superalloy has lost very little strength. At this temperature the yield strengths of the two alloys are about equal but the ductility of the tool steel is inferi-

TABLE IV

Specimen	Testing temp.,	Rahard- ness	Compressive yield strength, 0.2% offset	pressive	Percent deform. at max. strength	65
A-1	R.T. R.T. 1,000 1,000	57. 5 64	255, 000 355, 000 243, 000 253, 000	314,000 410,000 302,000 312,000	4. 1 2. 2 3. 8 2. 3	70

Note,—A-1, A-2: warm-worked specimens; M-1, M-2: fully heat treated M-50 tool steel.

The embrittling effect that the common platelike sigma produces has earned it a bad reputation in many alloy systems. However, the successful modification of the sigma phase by 75

the present invention has revealed that not only may the detrimental effects of sigma be alleviated in the superalloys, but also significant improvements may be provided therefrom. The presence of substantial quantities of dispersed, equiaxed sigma in the superalloys may be utilized to enhance strength and to provide high hardness without embrittlement. This suggests that these modified alloys may have utility as high temperature bearing alloys for, while high speed tool steels are rarely used in bearing applications above 600° F. and are limited to temperatures below 1,000° F., the equiaxed sigma superalloys are metallurgically stable to temperatures well over 1,000° F.

The significance of the invention is in fact considerably broader than a strengthening or hardening improvement may indicate. Alloy formulations may now be made without concern over any detrimental effects incident to sigma phase precipitation. Furthermore, not only may the sigma phase be utilized in an advantageous manner in the conventional sense but also, since its chemistry is variable, by changing its chemical makeup, control over its precipitation temperature can be exercised as necessary to tailor the alloy to a particular operating or processing regime. Still further, the controlled precipitation of the sigma phase or other topologically closepacked phases can now be utilized to advantage in the cobaltbase and austenitic iron-base alloys. In practical terms, therefore, a broad new field of alloy chemistry has been opened to practical utilization.

One significant advantage of the development, in addition 30 to the strengthening effect, is of fundamental interest to the gas turbine engine industry. Because the tendency for the formation of sigma is no longer a factor in alloy composition, high aluminum and titanium contents can be used resulting in very low alloy densities. High aluminum and titanium contents promote copious precipitation of γ' which enriches the γ matrix in sigma forming elements. The density of the principal tested alloy, for example, was 0.282 pounds per cubic inch which is lower than nearly every other nickel-base superalloy except IN-100 which is also sigma prone.

Although this invention has been described with great particularity and with reference to specific materials, processing parameters and examples for the purposes of illustration, the invention in its broader aspects is not limited to the specific details described, for obvious modifications will occur to those skilled in the art.

What is claimed is:

1. The method of processing the superalloys subjected to the precipitation of intermetallic compounds including a γ which comprises the steps of:

heat treating the alloy, including the step of aging above the minimum recovery temperature of the alloy, to establish a microstructure having the γ' precipitate in a stable homogenous distribution having an effective interparticle spacing not exceeding about 5 microns and a volume fraction of the precipitate not less than about 25 percent at the working temperature;

warm-working the alloy to effect a reduction in area of at least 15 percent while maintaining essentially the same γ' phase morphology established in the prior heat treatment, providing a microdefect array of regular geometry; and

heat treating the alloy at a temperature not exceeding the working temperature to precipitate the γ' phase remaining in solution and to precipitate any topologically closepacked phases as globular particles, stabilizing the microdefect array and providing additional strengthening.

2. The method according to claim 1 wherein:

the initial heat treatment includes aging at a temperature sufficiently above the minimum recovery temperature of the alloy to promote formation upon warm-working of the regular microdefect array comprising a polygonal subcell structure.

3. The method according to claim 1 wherein:

the initial heat treatment includes aging at a temperature sufficiently close to the minimum recovery temperature of the alloy to promote formation upon warm-working of the microdefect array comprising a warm-worked metallurgical substructure consisting of a randomly 5 nonoriented homogenous dislocation distribution.

4. The method according to claim 2 wherein:

the aging in the initial heat treatment is also above the precipitation temperature for the topologically closepacked phases.

5. The method according to claim 3 wherein:

the aging in the initial heat treatment is also above the precipitation temperature for the topologically closepacked phases.

6. The method of processing the nickel-base superalloys of 15 the γ - γ' type having a quantity of the γ' phase at room temperature exceeding about 25 volume percent and, optionally, a sigma phase precipitate which comprises the steps of: heat treating the alloy to solution the precipitate;

aging the alloy to precipitate the γ' phase to a minimum of 20about 25 volume percent in a stable homogenous distribution having an effective interparticle spacing not exceeding about 5 microns at a temperature sufficiently high to prevent substantial sigma phase precipitation;

warm-working the alloy to effect an area reduction of at 25 least 15 percent while maintaining essentially the same volume percent and distribution of the γ' phase established in the aging process, providing a microdefect

array of regular geometry; and

stabilizing the microdefect array of the alloy and providing 30 additional strengthening in a final heat treatment at a temperature not exceeding the prior aging and working temperature by precipitation of an additional quantity of the γ' phase and by precipitation of any sigma phase as 35 equiaxed particles.

7. The method according to claim 6 wherein:

the alloy is worked about the aging temperature. 8. The method according to claim 6 wherein:

the alloy is worked to effect an area reduction of 15-60 per-

9. The method according to claim 6 wherein:

the alloy is aged at a temperature sufficiently above the minimum recovery temperature of the alloy to promote formation upon warm-working of the regular microdefect 45 array comprising a polygonal subcell structure.

10. The method of strengthening the nickel-base superalloys of the γ - γ' type having a quantity of the γ' phase exceeding about 25 volume percent which comprises the steps of:

heat treating the alloy to solution at least the major portion 50 of the y' precipitate;

aging of the alloy at a temperature between the solvus temperature of the γ' phase and the minimum recovery temperature to reprecipitate at least 25 volume percent of the γ' phase, based on the overall alloy composition, in a sta- 55 ble uniform distribution at an effective interparticle spacing not exceeding about 5 microns;

working the aged alloy at about the aging temperature to effect a deformation corresponding to at least a 15 percent area reduction while maintaining essentially the same 60 volume percent and distribution of the γ' phase established in the aging process, providing a regular array of microcrystalline imperfections; and

heat treating the alloy at a temperature not exceeding the temperature of aging and working to cause precipitation 65

of that portion of the γ' phase remaining in solution after aging and to cause, in those alloys containing more than 0.05 weight percent carbon, precipitation of intragranular carbides, to provide a thermally and mechanically stable array of microcrystalling imperfections.

11. The method according to claim 10 wherein:

aging is performed at a temperature sufficiently above the minimum recovery temperature of the alloy to promote formation upon working of a polygonal metallurgical substructure having the dislocations aligned at subcell boundaries to provide a regular array of defects along these boundaries.

12. The method according to claim 10 wherein:

aging is performed at a temperature sufficiently close to the minimum recovery temperature for the alloy to promote formation upon working of a regular array of microcrystalline imperfections comprising a warmworked metallurgical substructure comprising a randomly nonoriented homogenous dislocation distribution.

13. The method of processing the nickel-base superalloys of the γ - γ' type enriched in those elements promoting the precipitation of topologically close-packed intermetallic phases which comprises the steps of:

heat treating the alloy to solution at least the major propor-

tion of the intermetallic precipitates;

aging the alloy at a temperature between the solvus temperature of the γ' phase and the minimum recovery temperature, and above the precipitation temperature of the topologically close-packed phases, to reprecipitate at least 25 volume percent of the γ' phase, based on the overall alloy composition, in a stable uniform distribution at an effective interparticle spacing not exceeding about 5 microns;

working the alloy at about the aging temperature to effect a deformation corresponding to at least a 15 percent area reduction while maintaining essentially the same volume percent and distribution of the γ' phase established in the

aging process; and

heat treating the alloy at a temperature not exceeding the temperature of aging and working to cause precipitation of that portion of the γ' phase remaining in solution after aging, precipitation of the topologically close-packed phases as equiaxed particles and, in those alloys containing more than 0.05 weight percent carbon, precipitation of intragranular carbides, to provide a thermally and mechanically stable array of microcrystalline imperfec-

14. The method according to claim 13 wherein:

aging is performed at a temperature sufficiently above the minimum recovery temperature of the alloy to promote formation of a polygonal metallurgical substructure having the dislocations aligned at subcell boundaries to provide a regular array of defects along these boundaries.

15. The method according to claim 13 wherein;

aging is performed at a temperature sufficiently close to the minimum recovery temperature for the alloy to promote formation of warm-worked metallurgical substructure comprising a randomly nonoriented homogenous dislocation distribution.

16. The method according to claim 13 wherein:

the final heat treatment includes a sequential heat treatment, one heat treatment being selected to precipitate the topologically close-packed phases and another being selected to precipitate the γ' phase.