NASA TECHNICAL MEMORANDUM

NASA TM X-71833

NASA TM X-71833

(NASA-TM-X-71833) HEAT TREATING OF A LAMELLAR EUTECTIC ALLOY (GAMMA/GAMMA PRIME + DELTA) (NASA) 22 p HC $3.50 CSCL 11F

N76-17231

Unclas

G3/26 14159

HEAT TREATING OF A LAMELLAR EUTECTIC ALLOY ($\gamma/\gamma' + \delta$)

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TECHNICAL PAPER to be presented at
International Gas Turbine Conference sponsored
by the American Society of Mechanical Engineers
New Orleans, Louisiana, March 21-25, 1976

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E-8531

Abstract

Eutectic superalloys are being developed at several laboratories for application as aircraft gas turbine airfoils. One such alloy $\gamma/\gamma' + \delta$ was subjected to several heat treatments to determine if its mechanical properties could be improved. It was found that by partially dissolving the γ' at 1210°C and then aging at 900°C the tensile strength can be increased about 12 percent at temperatures up to 900°C. At 1040°C no change in tensile strength was observed. Times to rupture were measured between 760 and 1040°C and were essentially the same or greater than for as-grown material. Tensile and rupture ductility of the alloy were reduced by heat treatment.

Introduction

A history of gas turbine development could well be titled "A Search for Higher Temperatures and Higher Pressures." As we are all aware this search has met much success in the past and still continues. The past successes have been due to a great degree to a combination of innovative turbine cooling systems and the development of the class of alloys commonly referred to as "superalloys."

The most advanced form of superalloys under development for use as gas turbine airfoils are the directionally

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solidified eutectic superalloys. They hold forth the promise of extending the useful metal temperature 50 to 100°C over more conventional superalloys. One such alloy is the $\gamma/\gamma' + \delta$ eutectic alloy having a nominal composition 20 w/o niobium-6 chromium-2.5 aluminum- balance nickel.

In the past significant improvements in mechanical properties of conventional superalloys have been obtained by using empirically developed thermal treatments. This investigation was initiated to determine if improvements in mechanical properties of a directionally solidified $\gamma/\gamma' + \delta$ eutectic alloy could be achieved by using thermal treatments similar to those used for conventional superalloys.

<u>Materials</u>

The materials studied were purchased from Pratt and Whitney Aircraft and are identical to those used by Gray in reference 1 and o[illegible] portions of his work cited later. They were right cylinders 8 cm long and 1.3 cm diameter having a nominal composition of 20 w/o Nb-6 Cr-2.5 Al-balance Ni. They were directionally solidified at 3 and 4 cm/hr in a furnace having a thermal gradient at the liquid solid interface in excess of 200°C/cm. The microstructure of the as-grown material is shown in figure 1. The structure consists of alternate lamellae of an intermetallic compound $\delta(Ni_3Nb)$ and γ Ni-base alloy in which γ' (Ni_3Al) has precipitated. The two phase γ/γ' lamellae resemble conventional superalloys in microstructure. The lamellae seen in figure 1(b) grew parallel to the solidification direction. These specimens contained a maximum of 25 per cent of the cellular or non-aligned regions as shown in figure 1.

<u>Mechanical Testing</u>

Preliminary tensile and stress rupture tests were run on cylindrical specimens having tapered end grips and a 0.63 cm diameter, 3.1 cm long gage section. Because Gray (reference 1) observed a large amount of scatter in stress rupture testing the same alloy at temperatures from 760 to 870°C using the same test specimen design, a different specimen design was used for the detailed stress rupture evaluation of a particular heat treatment. Based on Gray's work the specimen used for the detailed evaluation had threaded end grips and a 3.2 cm long gage section with a diameter of 0.5 cm. All tests were loaded parallel to the growth direction of the material. Tests were conducted in accordance with appropriate ASTM recommended practices. Single tests were used in the screening evaluation and duplicate tests were performed for the detailed evaluation of the selected heat treatment.

<u>Heat Treatment Selection</u>

Initially the γ' solvus temperature was determined by heating specimens for 4 hours at 1210, 1220, and 1230°C in argon followed by air quenching to room temperature. Photomicrographs of the resulting structures are shown in figure 2. Figures 2(a) and 2(b) show decreasing amounts of coarse undissolved γ'. Figure 2(c) shows complete dissolution of γ'. The γ' solvus was therefore taken as being between 1220 and 1230°C. This temperature is significantly higher than the 1149 to 1177°C reported in reference 2 (p. 22) for an alloy having 19.7 w/o Nb. It may also be seen by comparing figures 2(c) and 1(a) that there is no evidence of alteration of the basic lamellar structure even after heating to a temperature within 15°C of the reported 1242°C soidus temperature (ref. 2, p. 23).

Three material conditions, as-directionally solidified, solution treated 4 hours at 1225°C and partial solution treated for 4 hours at 1210°C were selected for aging. The solution treatments and aging treatments were performed under flowing argon and the material was air quenched to room temperature after all heat treating steps. The two conditions, as directionally solidified and solution treated at 1225°C, were selected as the limiting conditions. The partial solution was thought to be more indicative of a practical maximum temperature, being only about 30°C below the reported solidus temperature.

The aging response of as-grown and of γ' solutioned material was first screened metallographically. Aging times ranged from 8 to 48 hours and aging temperatures were varied from 750 to 1120°C. The results of these screening studies are summarized in Table I. Based on these observations it was decided to evaluate the effect of aging at temperatures of 900 and 1100°C on 870°C-515 MN/m² stress rupture life and 925°C tensile strength. The aging temperature of 900°C was selected, because it was the lowest temperature where significant γ' coarsening was observed. 1100°C was selected to represent an overaging condition and to be indicative of temperatures achieved in potential coating cycles.

The effect of the heat treatments on the 925°C tensile strength is shown in figure 3. The strength varied from 800 MN/m² for the partial solution plus 1100°C age condition to 960 MN/m² for the full solution condition. The solution treated and partial solution treated materials were stronger than as-grown material for both unaged specimens and those aged at 900°C. The average strength for material aged after solution and partial solution treatment was 892 MN/m², and for material aged directly after growth the strength was 857 MN/m². Material aged at 1100°C had an average strength of 833 MN/m² which was lower than the average strength of 903 MN/m² for both 900°C-aged and unaged material. The tensile elongation varied from 2 to 5 per cent, but no correlation with heat treatment condition was noted.

The effect of heat treatment on 870°C-515 MN/m² rupture life is shown in figure 4. The most apparent point is that aging increased the rupture life compared to unaged material. The average life of aged material was 292 hours, while the average life of unaged material was only 112 hours. The longest rupture lives were observed for partially solutioned and aged material. It also appears that aging at 1100°C gave slightly greater rupture lives than aging at 900°C. The average life for 1100°C aging was 304 hours compared to 281 hours for material aged at 900°C. Rupture elongations varied from 6 to 10 per cent, but could not be correlated with heat treatment effects.

The microstructural feature which appears to influence the tensile strength is the γ' size. The higher temperature aging treatments, which resulted in larger γ' sizes, lower strength. The fracture modes did not appear to change with heat treatment. A typical elevated temperature tensile fracture is shown in figure 5. The fracture is primarily trans-granular with some shearing of grain boundaries parallel to the growth direction. Secondary cracking appears to form first in δ lamellae along twins. Cooperative twinning of δ and deformation in γ/γ' lamellae result in kinking in the lamellar structure. The kinking is assumed to be a precursor to cracking and fracture. Portions of the fractured surface are therefore seen to be parallel to the twin directions and kinking direction in the alloy.

Typical photomicrographs of the directionally solidified eutectic alloy which has been partially solution treated and aged are shown in figure 6. Comparing figures 6(a) and 6(b) it can be seen that aging at 1100°C produces a coarser γ' than does 900°C aging. The bimodal γ' size distribution resulting from the partial solution treatment is more apparent in figure 6(b). It was common in this investigation to see γ' dissolve more completely in the cellular regions than in lamellar regions as can be seen in figure 6(b). This phenomenon is presumed to occur because the cellular regions are the last to solidify and therefore should have higher concentrations of solutes than the lamellar regions. In this case it is believed that an increase in Cr concentration is locally lowering the γ' solvus temperature. Although it is not apparent from these photomicrographs, the cellular regions were observed to contain more Widmänstatten δ phase than the lamellar regions.

Based on the previously discussed tensile and stress rupture results, the heat treatment consisting of partial solution treating at 1210°C followed by aging 24 hours at 900°C was selected for further evaluation of tensile and stress rupture strengths. The selected heat treatment appeared to offer the best balance of tensile strength and rupture life of the 9 conditions screened.

<u>Selected Heat Treatment</u>

The tensile strength of $\gamma/\gamma' + \delta$ which was partially solutioned at 1210°C and aged 24 hours at 900°C is shown in Table II(a) and compared to as-grown material (ref. 1) in figure 7. At temperatures below 1040°C the heat treated material is about 12 per cent stronger than the as-grown alloy. The average tensile strength of the heat treated material decreased from 1405 MN/m^2 at room temperature to 980 MN/m^2 at 925°C. At 1040°C the alloy had a tensile strength of about 650 MN/m^2 for both the heat treated and as-grown conditions.

At room temperature and 760°C the elongation of the heat treated eutectic was slightly lower than for the as-grown alloy. At 925 and 1040°C the elongation was essentially the same for both the heat treated and as-grown alloy. It appears that for temperatures below the aging temperature of 900°C the heat treatment is beneficial to strength, but adversely affects tensile elongation.

Duplicate stress rupture tests were run at temperatures from 760 to 1040°C at stresses from 760 to 150 MN/m^2. The results of these tests are listed in table II and are shown on a Larson-Miller plot in figure 8. Also shown in figure 8 as a dashed curve are the results of threaded end test-bar tests of the as-directionally solidified alloy (private communication with H. R. Gray, NASA Lewis Research Center). The stress rupture strength of the heat treated alloy can be seen to compare well with results from the as-grown material. Except for a single short life data point of 318.5 hours at 800°C the life of the heat treated alloy seems to be greater than that of the as-grown alloy at temperatures of 760 and 800°C. The rupture ductility of the heat treated material in this investigation was generally lower than that observed by Gray.

Discussion

This work has shown that modest improvements in tensile strength up to the aging temperature and perhaps of rupture life up to 800°C can be achieved in $\gamma/\gamma'+\delta$ by the application of a simple heat treatment. The strength values measured in this investigation are even greater than those reported in reference 3 for the same alloy grown at about twice the solidification rate used in this investigation. We suggest that the improvement in strength is related to the finer γ' size achieved by heat treating compared to the as-grown alloy. The reasons for the apparent differences in mechanical properties between the partial and full solution treated materials are not understood.

Work by others on this alloy system has noted low shear strength parallel to the lamellar growth direction (ref. 4) and poor transverse ductility (ref. 5) and rupture strength. It is therefore interesting to speculate whether the heat treatments used here might also improve shear strength or transverse properties. Because the most significant microstructural change noted in this investigation appears to be γ' size distribution and because crack propagation for off-axis loading has been shown to be along cell walls and grain boundaries (ref. 4) it is not expected that the heat treatments in this investigation should influence off-axis properties. However, examination of our data in the light of Gray's work (ref. 1) suggests a promise of improved off-axis properties. Gray observed large stress rupture data scatter when he used tapered head test specimens. We believe that his data scatter was caused by off-axis loading created by alignment problems associated with his test configuration. In figure 4, where we used the tapered head test configuration the poorest life for an aged sample was almost twice the life of the best unaged sample. At the same test of 870°C at a stress of 515 MN/m^2 in figure 8, it can be seen that the aged alloy has essentially the same life as Gray (private communication) observed for as-grown material where both investigations used threaded end grips. Also note that the test in figure 4 having the same heat treatment of 4 hours at 1210°C followed by aging 24 hours at 900°C, and using tapered end grips, had essentially the same rupture life of about 350 hours as the data listed in table II(b) and plotted in figure 8 where threaded end test bars were used. One then concludes that the aged material above 800°C is not significantly different from the unaged alloy when both are tested with good axial alignment.

The reason for the apparent improved properties observed for aged material in our screening studies still needs attention. We suggest that the aging treatment may reduce the sensitivity of the alloy to off-axis loading thereby causing the aged material to appear better in stress rupture tests using the tapered head specimen. Perhaps the transverse ductility is improved. The effect of the selected heat treatment on transverse ductility is currently being investigated by Pratt and Whitney Aircraft as part of a NASA funded program under contract NAS 3-17811.

Summary

An investigation was conducted to determine if mechanical property improvements could be achieved in a directionally solidified $\gamma/\gamma'+\delta$ eutectic alloy by applying heat treatments similar to those used for conventional Ni-base superalloys. The alloy had a nominal composition of 20w/o Nb- 6 Cr- 2.5 Al- balance Ni. The more significant findings of this investigation are as follows:

1. The best combination of 925°C tensile and 870°C stress rupture properties resulted from a heat treatment consisting of a partial solution treatment of 4 hours at 1210°C followed by aging 24 hours at 900°C. For temperatures below 925°C the heat treated material was about 12 per cent stronger than for as-grown material. The tensile strength resulting from this heat treatment decreased from 1405 MN/m^2 at room temperature to 650 MN/m^2 at 1040°C. At 760 and 800°C the heat treated alloy appears to have a greater stress rupture strength than as-grown material. For temperatures from 870°C to 1040°C the stress rupture strength of heat treated and as-grown material appear to be equivalent. The ductility of the heat treated material was generally less than that reported for as-grown material.
2. The γ' solvus temperature for the alloy having a nominal composition 20w/o Nb- 6 Cr-2.5 Al- balance Ni was determined metallographically to be between 1220 and 1230°C.
3. For material solution treated near the γ' solvus temperature, aging at 900°C resulted in greater 925°C tensile strength than aging at 1100°C.

4. Longer stress rupture lives at 870°C with a stress of 515 MN/m^2 were obtained from aged samples which had been partially solution treated at 1210°C than for material which had been fully solution treated at 1220°C or aged directly after being grown.

5. Aging at 900 and 1100°C appeared to increase the rupture life at 870°C with a stress of 515 MN/m^2. It is postulated that this apparent improvement, which was obtained when tapered end test bars were used, may be indicative of improvements in the transverse properties of the alloy.

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TABLE I. - THERMAL TREATMENTS AND MICROSTRUCTURAL OBSERVATIONS[a]

Thermal treatment		Microstructural observation	
		Material condition	
Aging temperature, °C	Aging time, hr	As directionally solidified	γ' solutioned (4 hr/1225° C - A.Q.)
	Zero (no aging)	Cuboidal γ' (0.4 μm) & disk shaped γ''[b] in γ; occasional lath shaped δ in cellular γ	Cuboidal γ' (0.15 μm) in γ
750	16 & 48		
800	16 & 48		Cuboidal γ' (0.14 μm) in γ
850	8 & 24		Cuboidal γ' (0.16 μm) in γ
900	8 & 24	γ in δ & δ in γ/γ' (more in cellular regions; γ' in γ coarsens and becomes spherical as aging temperature increases	Cuboidal γ' (0.18 μm) in γ & γ'' in γ
950	8 & 24		Cuboidal γ' (0.29 μm) in γ; γ'' in γ; γ in δ & δ in γ/γ' (more in cellular regions)
1040	8		Cuboidal γ' (0.29 & 0.38 μm) in γ; γ'' in γ; δ in γ and less γ in δ (more in cellular regions)
1120	8		Spheroidal γ' (0.48 μm) in γ; less and coarser γ in δ; more and coarser δ in γ/γ'
			Spheroidal γ' (0.68 μm) in γ; more and coarser δ in γ/γ'; less and coarser γ in δ

[a] All had alternate lamellae of γ/γ' and δ (less than 25 percent cellular region).
[b] γ'' is bct Ni_3Nb.

TABLE II. - TEMPERATURE DEPENDENCE OF THE STRESS-RUPTURE AND TENSILE PROPERTIES OF HEAT-TREATED* DS γ/γ'-δ EUTECTIC ALLOY.

(Growth rate 4 cm/hr)

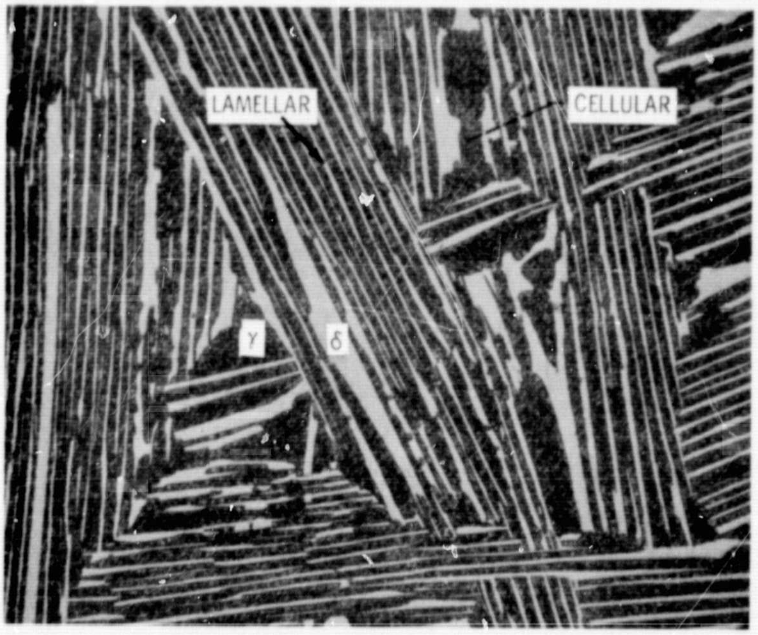
(a) Tensile properties

Temperature, ^{o}C	Tensile strength, MN/m^2	Elongation, percent	Reduction in area, percent
Room (23)	1400	1	3
	1410	4	3
760	1115	8	10
	1165	3	6
925	940	3	3
	1025	4	3
1040	655	13	20
	650	19	21

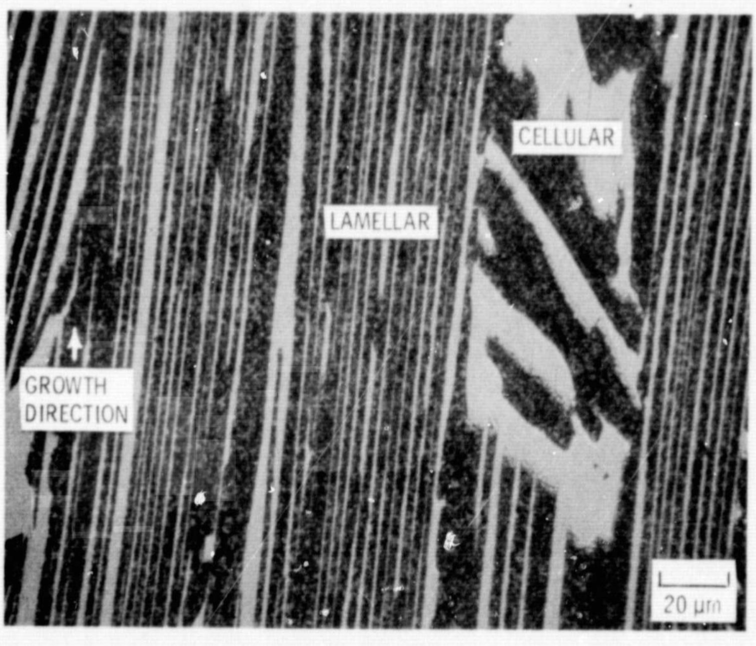
(b) Stress rupture properties

Temperature/ stress, $^{o}C/MN/m^2$	Rupture life, hr	Elongation, percent	Reduction in area, percent
760/760	> 3330		
	2182.0	1	3
800/690	1149.7	7	8
	318.5	2	5
870/515	324.0	8	8
	445.0	6	8
	308.3	5	9
1040/340	3.3	11	21
	4.5	15	18
1040/150	151.5	12	16
	234.8	12	16

*4 hr/1210° C -A.Q. - 24 hr/900° C - A.Q.



(a) TRANSVERSE TO GROWTH DIRECTION.



(b) LONGITUDINAL.

Figure 1. - Typical microstructure of as-grown $\gamma/\gamma' + \delta$ eutectic alloy. Etchant: 30 ml HNO_3, 30 ml H_2O, 30 ml CH_3COOH and 1 ml HF (mixed acids).

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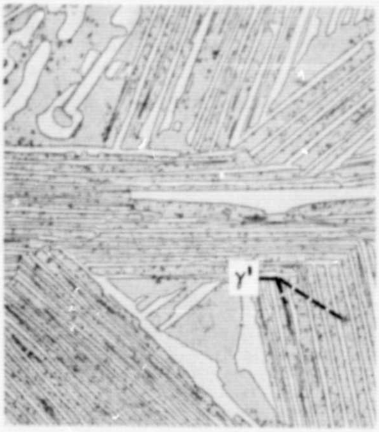
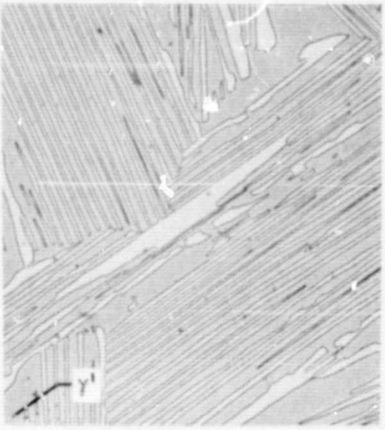
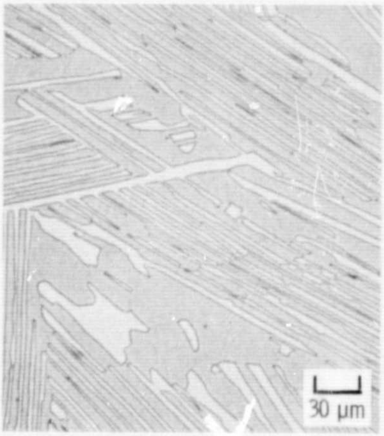


(a) 4 HOURS AT 1210° C.



(b) 4 HOURS AT 1220° C.



(c) 4 HOURS AT 1230° C.

Figure 2. - Determination of γ' solvus temperature for $\gamma/\gamma' + \delta$ (transverse sections; etchant: mixed acids).

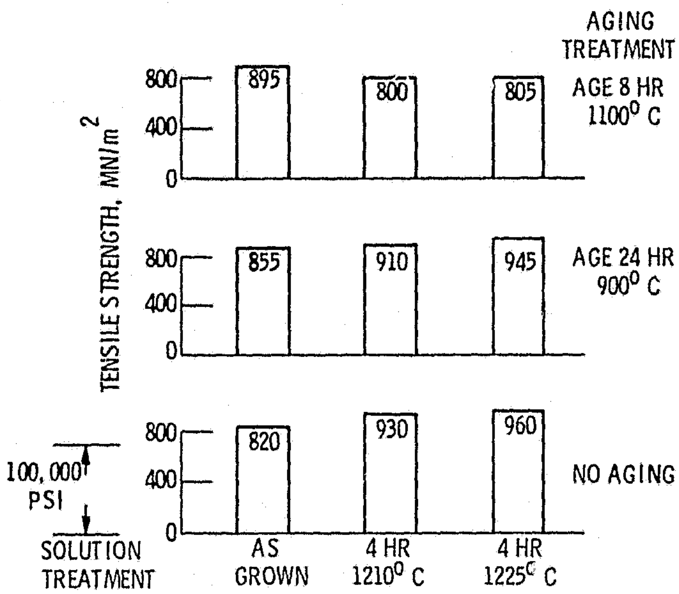


Figure 3. - Effect of heat treatment on 925° C tensile strength of $\gamma/\gamma' + \delta$.

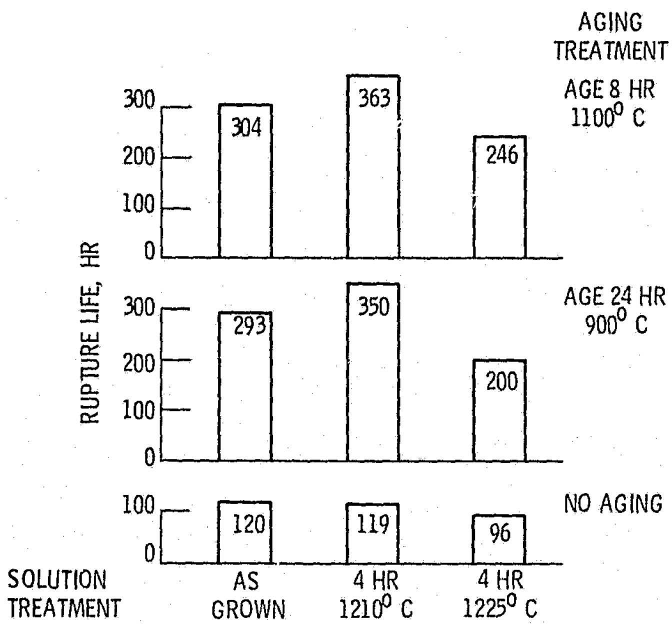


Figure 4. - Effect of heat treatment on stress rupture life of $\gamma/\gamma' + \delta$. (Tests at 870° C, 515 MN/m² (75,000 PSI.)

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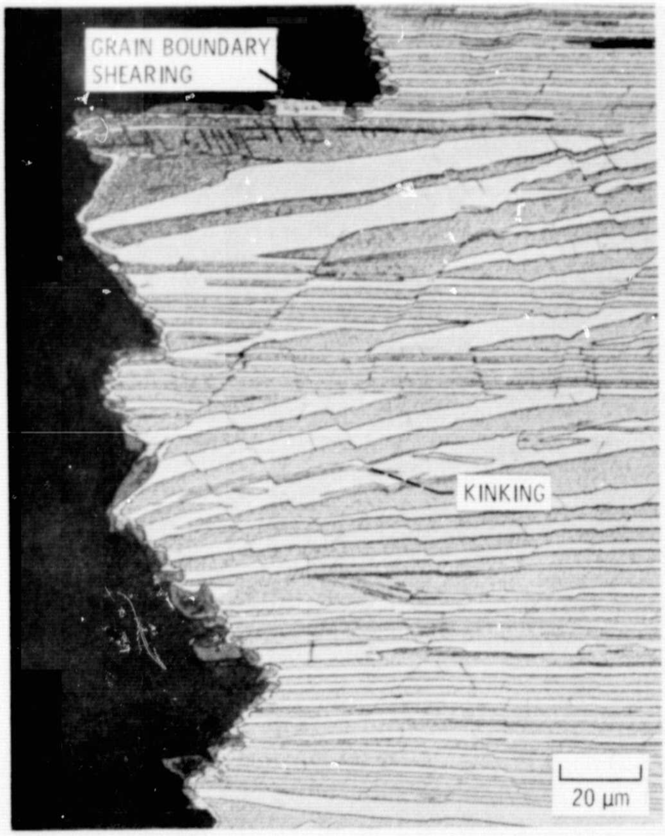
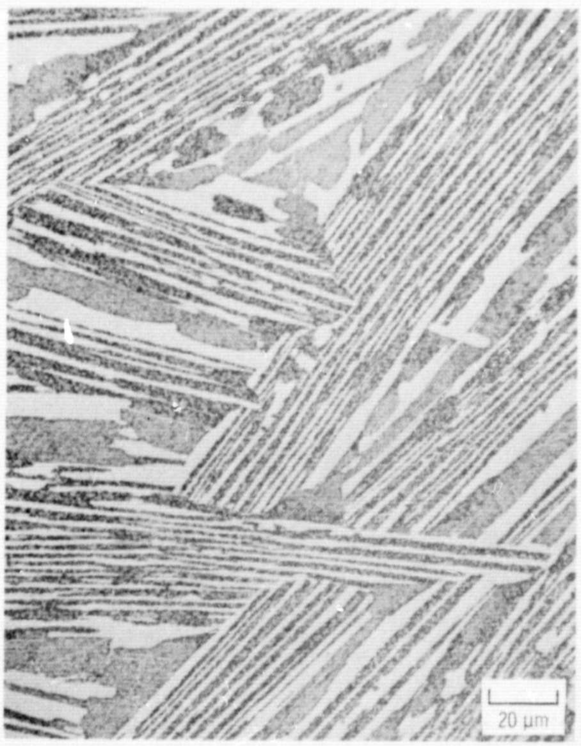


Figure 5. - Microstructural appearance of a typical tensile fracture (925° C). (Longitudinal section; etchant: mixed acids.)

(a) 8 HOURS AT 1100° C.



(b) 24 HOURS AT 900° C.

Figure 6. - Effect of aging treatments following partial γ' solution treatments (transverse sections; etchant: mixed acids).

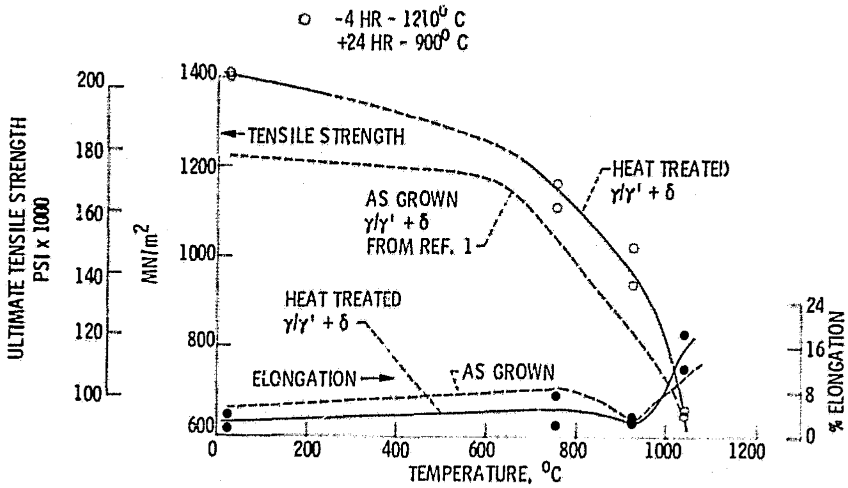


Figure 7. - Effect of temperature on tensile properties of γ/γ' + δ.

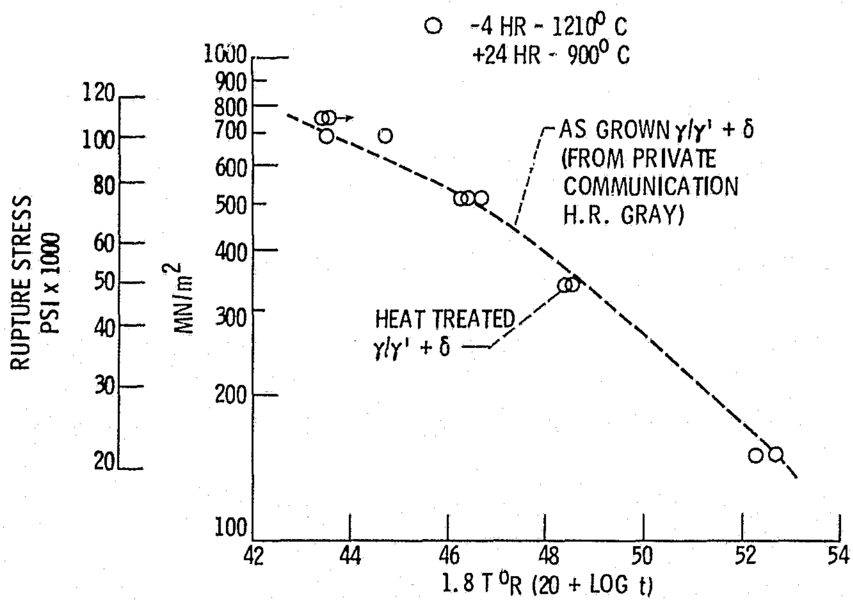


Figure 8. - Comparison of stress-rupture strength of as-grown and heat treated γ/γ' + δ.