PRECIPITATION BEHAVIOR IN AEREX™ 350

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Abstract

The precipitation behavior of a new nickel base superalloy, AEREX™* 350, is examined. An understanding of the microstructure as influenced by different heat treatments is necessary in order to realize the full potential of the alloy. This paper presents the effects of solution treating and aging treatments on the resulting microstructure. Samples are solution treated at 1093°C (2000°F) or 1052°C (1925°F) and then given various aging treatments. Optical and scanning electron microscopy are used to examine the phases in the microstructure. The precipitates are examined in the scanning electron microscope. In addition, the relationship between the microstructure and mechanical properties are elucidated.

Introduction

AEREX™ 350 is a new nickel-base superalloy with a nominal composition of 25 wt% cobalt, 17 wt% chromium, 3 wt% molybdenum, 2 wt% tungsten, 4 wt% tantalum, 2 wt% titanium, 1 wt% aluminum, 1.1 wt% niobium, 0.015 wt% carbon and 0.015 wt% boron. The alloy was designed to provide higher temperature strength than the currently available wrought superalloys and still maintain workability for ease of manufacturing. Initial property studies have shown that the alloy has superior properties for applications requiring operating temperatures of 704°C (1300°F) or higher.[1] It has been shown that, in the cold worked condition, the alloy can be used for fastener applications up to 732°C (1350°F).[2] Figure 1 illustrates the high temperature strength of AEREX™ 350 in a solution treated and aged condition as compared to other solution treated and aged superalloys.[3,4,5,6,7]

The composition of AEREX™ 350, predicts that its strengthening is derived from gamma prime (γ') and solid solution strengthening. The low "aluminum + titanium" level also predicts that the alloy is readily hot-workable. Forging and hot rolling trials done at Latrobe Steel Company have confirmed this. Preliminary studies performed at Micro-met Laboratories, Inc. (utilizing x-ray diffraction on extraction samples) have identified the presence of the hcp phase, eta, Ni_3Ti (η).[8] This phase is common in nickel-iron base superalloys such as alloys 901 and 706 and iron-base alloys such as A-286.[9] Further studies including x-ray diffraction and transmission electron microscopy (TEM) have also verified the presence of both η and γ'.[10,11] It is the purpose of this paper to illustrate how heat treatment may effect the microstructure and how this, in turn, has an effect on the properties.

Procedure

Samples of hot rolled AEREX™ 350 were solution treated using either a 1093°C (2000°F) or 1052°C (1925°F) temperature for 1 hour in an air atmosphere furnace and water quenched. Samples from each solution treatment were aged for 4 hours at temperatures ranging from 732°C (1350°F) to 899°C (1650°F) and then air cooled to room temperature. Several samples were also heat treated using a double age (more commonly known as a stabilize and age heat treatment) to determine the effects on hardness and microstructure. These samples were then electropolished in a 20% sulfuric acid solution at 25 volts and electroetched in a solution consisting of 15g of chromium trioxide in 170ml of phosphoric acid and 10ml of sulfuric acid at 6 volts. The resulting microstructures were examined in the scanning electron microscope (SEM).

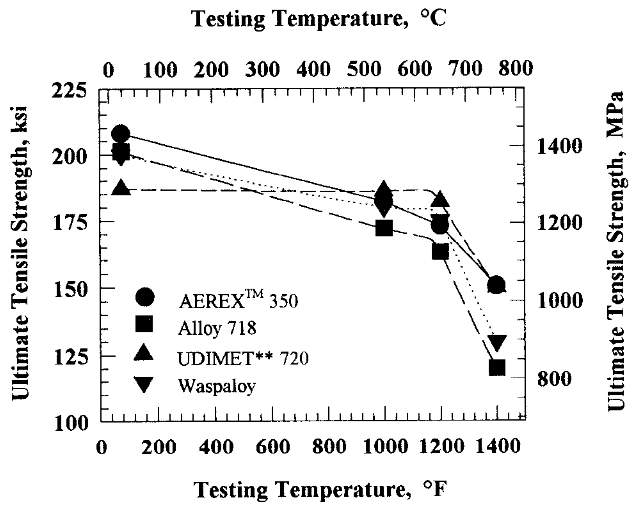

Figure 1. Tensile strength of selected superalloys.

* AEREX™ is a trademark of SPS Technologies, Inc.
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Superalloys 1996
Edited by R. D. Kissinger, D. J. Deye, D. L. Anton,
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At lower aging temperatures, γ' could not be resolved in the SEM. The observed increase in hardness, however, indicates that precipitation of a strengthening phase has occurred. Samples solution treated at 1093°C (2000°F) were aged at 732°C (1350°F) for longer times to reveal the precipitate. A sample aged for 64 hours was examined in the SEM. After reviewing the microstructure and related hardnesses of the heat treated samples, several heat treatments were chosen for further evaluation. Room and elevated temperature tensile tests were selected to study the relationship between mechanical properties and microstructure.

Results

Heat Treatment

Hardness measurements of solution treated and aged samples of AEREX™ 350 (composition illustrated in Table 1) are tabulated in Table II. Aging the solution treated samples provides a notable increase in hardness. The aging curves for both solution treatment temperatures are illustrated in Figure 2. A double aging treatment also contributes to the hardening response.

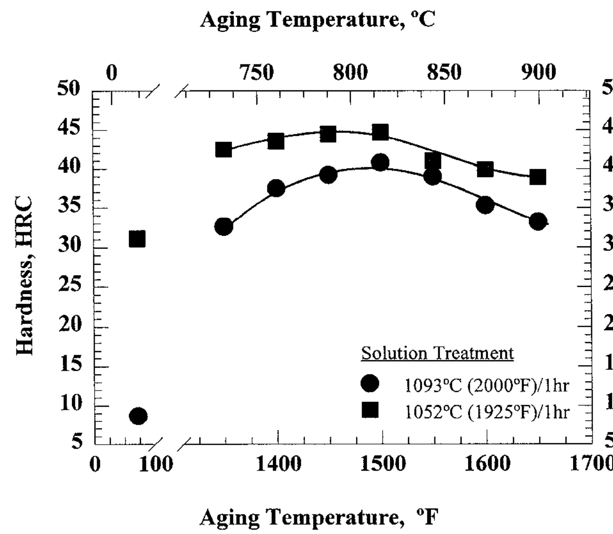

Figure 2. Aging curve for AEREX™ 350.

TABLE I. Composition of AEREX™ 350 - Heat G2389

	Ni	Co	Cr	Mo	W	Ti	Al	Ta	Nb	C	B	S	P
Ingot 1	44.69	25.15	17.03	3.05	2.17	2.07	0.84	4.02	1.13	0.019	0.021	0.001	0.004

TABLE II. Hardness Results (HRC) from Heat Treated Samples of AEREX™ 350

Aging Temperature (4hr/Air Cool)	Solution Treatment 1093°C (2000°F) (1hr/WQ)	Solution Treatment 1052°C (1925°F) (1hr/WQ)
As Solutioned	8.7	31.1
732°C (1350°F)	32.6	42.5
760°C (1400°F)	37.5	43.6
788°C (1450°F)	39.2	44.5
816°C (1500°F)	40.8	44.7
843°C (1550°F)	39.0	41.0
871°C (1600°F)	35.3	39.9
899°C (1650°F)	33.2	38.9
899°C (1650°F) + 732°C (1350°F)	40.0	42.7
899°C (1650°F) + 760°C (1400°F)	40.1	43.2
899°C (1650°F) + 788°C (1450°F)	39.9	42.4
899°C (1650°F) + 816°C (1500°F)	39.2	42.0
899°C (1650°F) + 843°C (1550°F)	36.8	40.1

Microstructure

Solution Treat. The microstructures obtained after solution treatment are shown in Figures 3 and 4. The grain size is fine (ASTM 8 or finer) in the samples solution treated at 1052°C (1925°F) and platelets of η appear at the grain boundaries (Fig. 3). Figure 4 shows the microstructure after solution treating at 1093°C (2000°F). The grains have grown (a grain size of ASTM 2, with an occasional 0) and the only apparent precipitates are primary carbides (MC, where M consists of combinations of tantalum, titanium and niobium).

Single Age. Aging at 732°C (1350°F) for 4 hours results in no apparent change in the microstructure when examined in the SEM (Figures 5 and 6). The hardness increase shown in Figure 2 illustrates, however, that some strengthening is occurring upon aging. Figure 7 shows a sample aged at 732°C (1350°F) after it has been aged for 64 hours; precipitates are now visible in the SEM.

SEM examination of samples heat treated at 788°C (1450°F) reveals a fine dispersion of γ' particles beginning to appear in the grain interiors. Small platelets of η are starting to precipitate at the grain boundaries in the 1093°C (2000°F) solution treated material

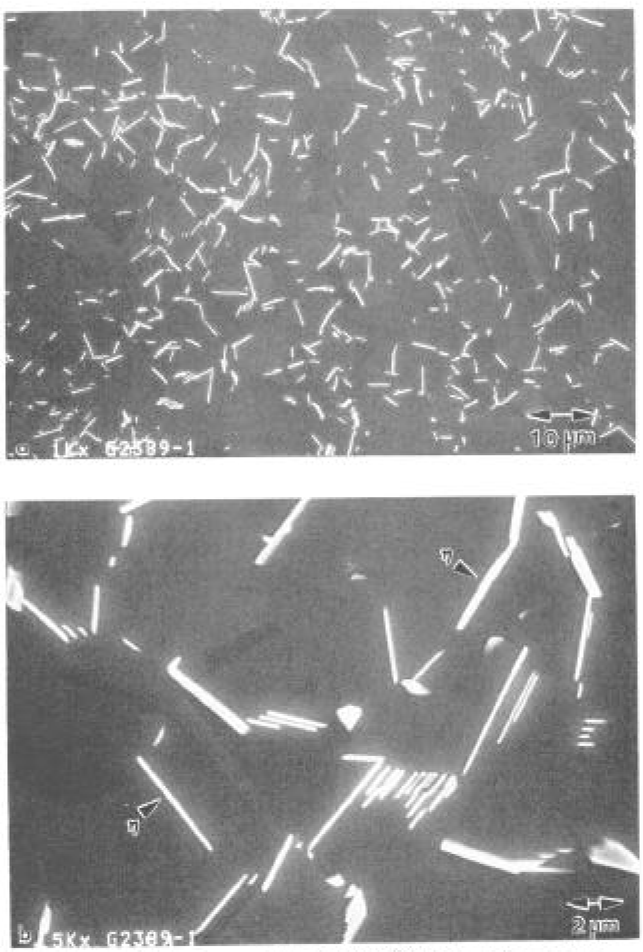

Figure 3. SEM micrograph of AEREX™ 350 solution treated for 1 hour at 1052°C (1925°F) illustrating the presence of eta (η) phase. (a) 1,000x and (b) 5,000x

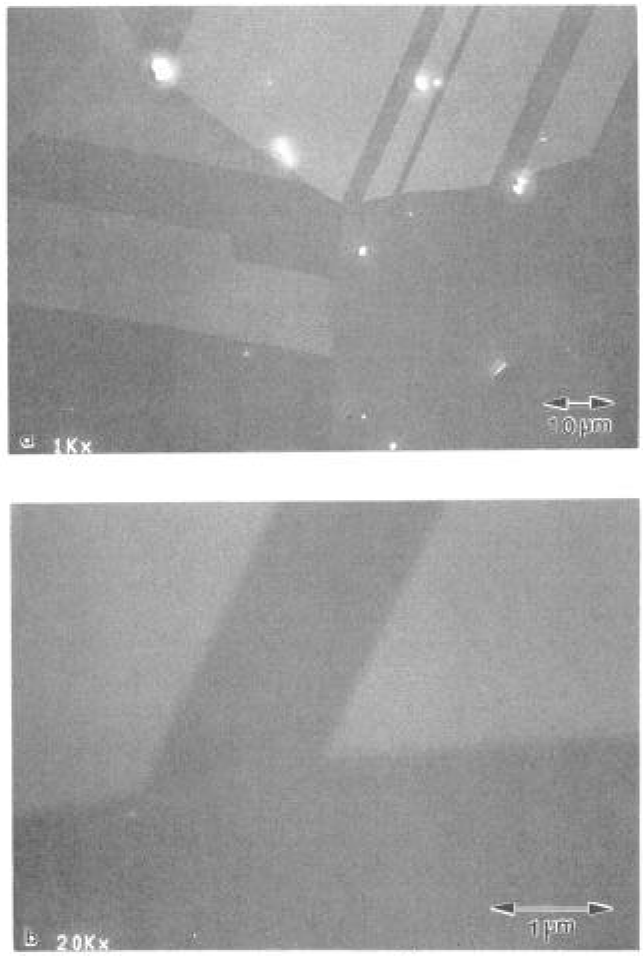

Figure 4. SEM micrograph of AEREX™ 350 solution treated for 1 hour at 1093°C (2000°F). (a) 1,000x and (b) 20,000x

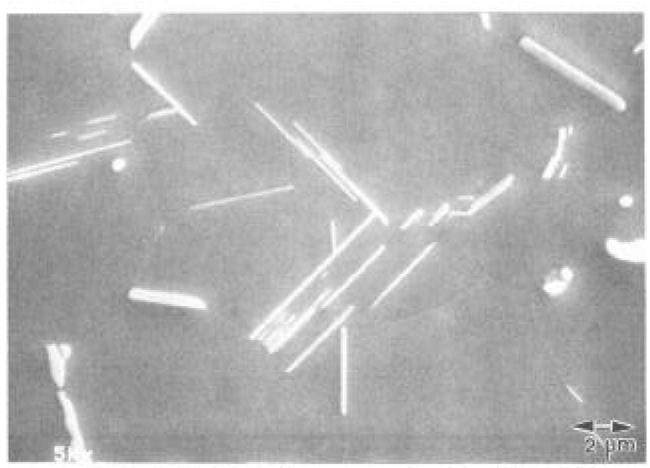

Figure 5. AEREX™ 350 solution treated at 1052°C (1925°F) and aged at 732°C (1350°F) for 4 hours (5000x).

as illustrated in Figure 8. Figure 9 shows the η phase is beginning to develop a Widmanstätten plate structure in the 1052°C (1925°F) solution treated sample aged at 816°C (1500°F). The γ' precipitates are now readily visible in the SEM.

Double Age. Figures 10 and 11 illustrate the effects of a double age. A sample solution treated at 1052°C (1925°F) and aged at 899°C (1650°F) for 4 hours plus 732°C (1350°F) for 4 hours is shown in Figure 10. The 1093°C (2000°F) solution treated sample aged at 871°C (1600°F) for 4 hours plus 760°C (1400°F) for 16 hours is shown in Figure 11. Samples with a double aging heat treatment reveal that the γ' precipitates have coarsened but remain evenly distributed. The η morphologies consist of Widmanstätten plate structures at the grain boundaries. Intragranular platelets of η are also apparent.

Mechanical Properties

The results from room temperature tensile tests are shown in Table III. The lower solution treatment temperature provides higher strength. Certainly, the smaller grain size has an influence on tensile strength. The Hall-Petch relationship, however, would predict a higher yield strength in the lower temperature solution treated material than is observed. This is determined by calculating a value for k in the Hall-Petch equation ($\sigma_{ys} = \sigma_o + kd^{-1/2}$). Given the yield strength in the high temperature solution treatment and an average grain size of 180 µm, the 22 µm grain size produced in the lower temperature solution treat would result in a very high yield strength. Although this is a very crude approximation which warrants further investigation, it illustrates that other mechanisms, such as precipitation, must be taken into consideration.

Table IV shows the elevated temperature tensile data. Once again, it is interesting to note the effect of precipitating phases on strength. Even of more interest is the ductility at elevated temperatures. It appears that the presence of η diminishes the ductility. The 1093°C (2000°F) solution treated material is more ductile. The effects of the solution treat temperature on ductility are illustrated in Figure 12. The ductility of AEREX™ 350 from the heat treatment providing the least ductility is compared to other superalloys in Figure 13.

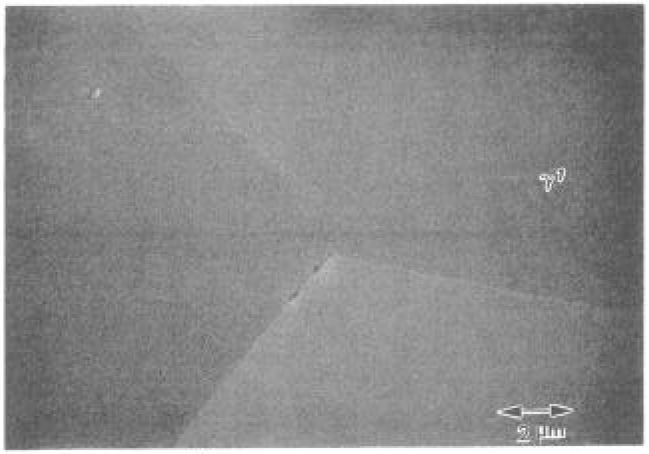

Figure 7. AEREX™ 350 solution treated at 1093°C (2000°F) and aged at 732°C (1350°F) for 64 hours (5000x).

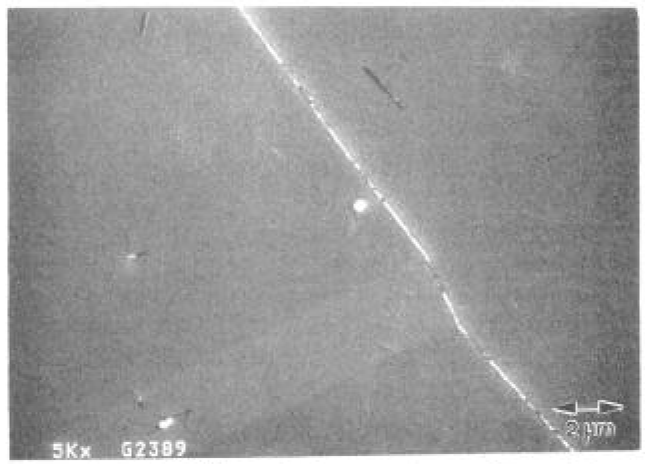

Figure 8. AEREX™ 350 solution treated at 1093°C (2000°F) for 1 hour and aged at 788°C (1450°F) for 4 hours (5000x).

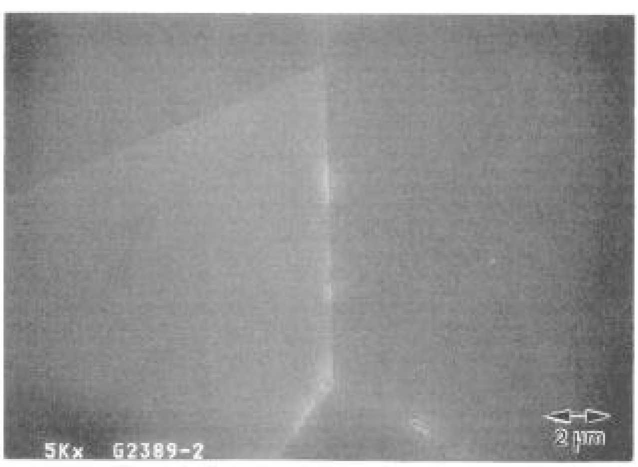

Figure 6. AEREX™ 350 solution treated at 1093°C (2000°F) and aged at 732°C (1350°F) for 4 hours (5000x).

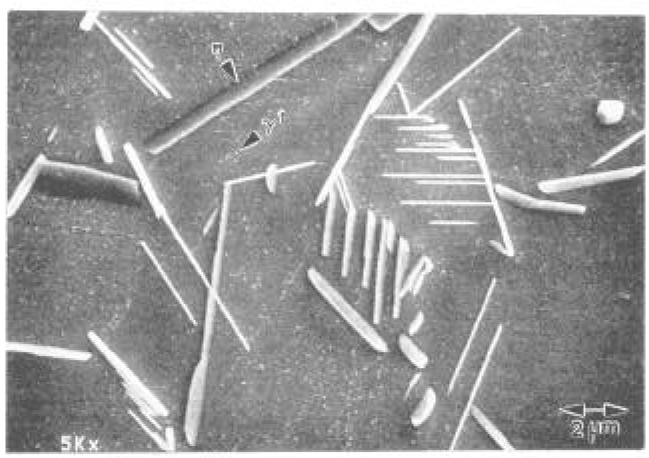

Figure 9. AEREX™ 350 solution treated at 1052°C (1925°F) for 1 hour and aged at 816°C (1500°F) for 4 hours (5000x).

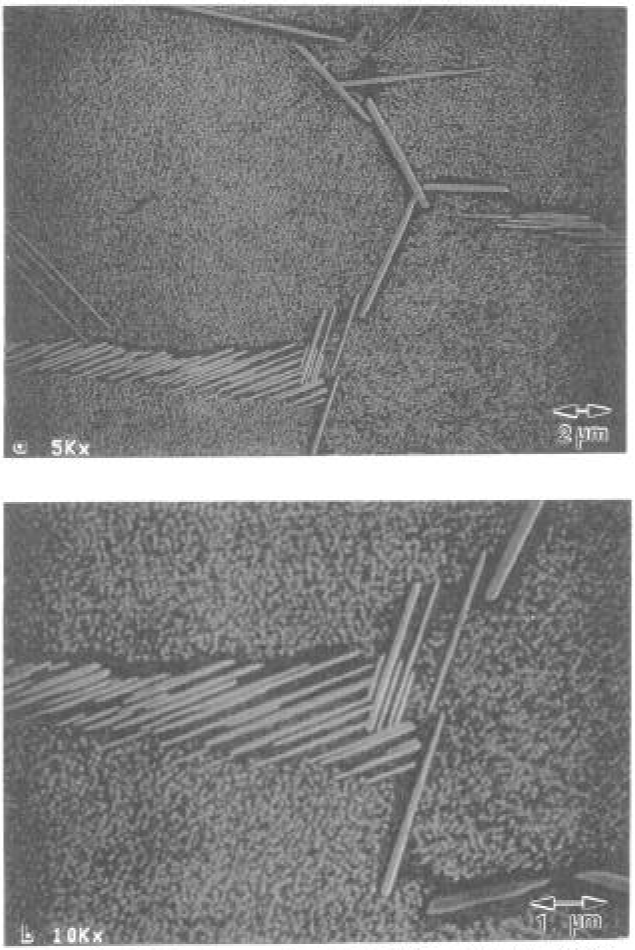

Figure 10. SEM micrograph at (a) 5,000x and (b) 10,000x illustrating AEREX™ 350 solution treated at 1052°C (1925°F) for 1 hour and aged at 899°C (1650°F) for 4 hours + 816°C (1500°F) for 4 hours.

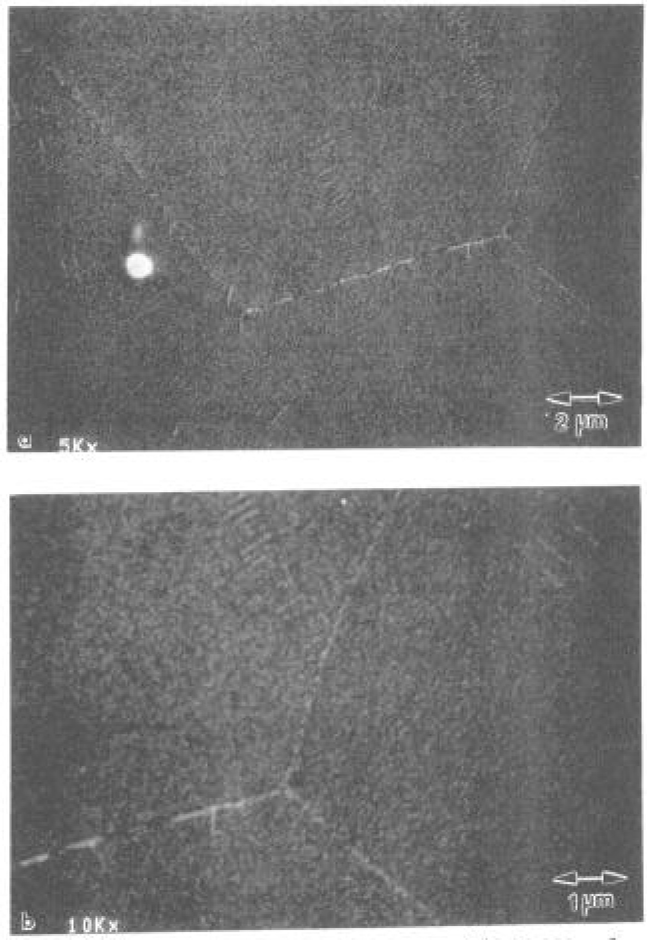

Figure 11. SEM micrograph at (a) 5,000x and (b) 10,000x of AEREX™ 350 solution treated at 1093°C (2000°F) for 1 hour and aged at 871°C (1600°F) for 4 hours + 760°C (1400°F) for 16 hours.

Table III. Room temperature tensile results and precipitates present in the microstructure. (Note: Carbides are present in all heat treated conditions. gb=grain boundary, w=Widmanstätten structure, intra=intragranular)

Heat Treatment (ST/Age)	Figure No.	γ'	η	UTS (MPa)	YS (MPa)	Elong (%)	R.A. (%)
1093°C/1h+732°C/4h	Figure 6	fine	none	1091.4	751.5	52.3	54.9
1093°C/1h+816°C/4h		small	fine gb	1301.7	947.3	40.0	45.6
1093°C/1h+899°C/4h		large	gb (w)	1266.5	812.9	43.8	39.0
1093°C/1h+871°C/4h+760°C/16h	Figure 11	coarse	gb (w)	1456.2	1042.5	31.5	37.8
1093°C/1h+899°C/4h+816°C/4h		coarse	gb(w)/intra	1361.7	879.8	28.5	26.3
1052°C/1h+732°C/4h		fine	gb	1499.6	1088.0	30.0	34.7
1052°C/1h+816°C/4h	Figure 9	small	gb (w)	1515.5	1133.5	27.8	32.5
1052°C/1h+899°C/4h		large	gb(w)/intra	1441.0	992.8	27.3	29.9
1052°C/1h+899°C/4h+816°C/4h	Figure 10	coarse	lg. gb/intra	1436.2	855.6	25.3	27.2

Discussion of Results

AEREX™ 350 has been shown to precipitate both γ' and η upon aging. The 1093°C (2000°F) solution treat temperature is above the eta solvus and the 1052°C (1925°F) is just below the eta solvus temperature. Samples from both solution treatments aged at 732°C (1350°F) are representative of an underaged condition. In the 1093°C (2000°F) solution treated sample, η is still not present and the ductility is at its greatest in both room and elevated temperature tensile tests. Material heat treated to the peak aging condition, which occurs at 816°C (1500°F) for both solution treatments, exhibits a maximum in both hardness and tensile strength. Overaging not only decreases strength, it decreases ductility as well.

Double aging increases the strength in the 1093°C (2000°F) solution treated material with a small sacrifice to ductility. The double age in the 1052°C (1925°F) solution treated material has very little effect on either strength or ductility. This behavior can be related back to the microstructure. The double age effectively coarsens the γ' for strengthening in the 1093°C (2000°F) solution treated material with a good balance of the η phase. The η phase in the 1052°C (1925°F) solution treated material is becoming too coarse and continuous. This morphology would not be beneficial to the alloy.

Eta phase is an ordered geometrically close packed (GCP)-type precipitate with an HCP crystal structure. The η phase is common in iron- and nickel-iron-base superalloys but is generally not present in cobalt base superalloys. Although AEREX™ 350 is a nickel base alloy, it contains a significant amount of cobalt. GCP phases do not form easily in cobalt-base alloys, although efforts have been made to generate GCP phases in cobalt alloys for additional strengthening.[12]

Table IV. Elevated Temperature Tensile Results

Heat Treatment (ST/Age)	Figure No.	Test Temp (°C)	UTS (MPa)	YS (MPa)	Elong (%)	R.A. (%)
1093°C/1h+732°C/4h	Figure 6	704	901.8	678.5	43.0	42.4
1093°C/1h+732°C/4h	Figure 6	760	791.5	538.5	40.0	42.8
1093°C/1h+816°C/4h		704	1094.9	790.8	22.5	28.4
1093°C/1h+871°C/4h		704	946.0	704.0	12.5	19.5
1093°C/1h+899°C/4h		704	884.6	721.2	9.0	15.6
1093°C/1h+871°C/4h+760°C/16h	Figure 11	704	1140.4	823.3	10.0	12.4
1052°C/1h+732°C/4h		704	1148.0	939.8	13.5	14.9
1052°C/1h+816°C/4h	Figure 9	704	1228.0	966.6	12.5	11.9
1052°C/1h+816°C/4h	Figure 9	760	1037.0	845.3	6.5	9.2
1052°C/1h+899°C/4h		704	1176.9	861.8	14.0	11.5
1052°C/1h+899°C/4h+816°C/4h	Figure 10	704	1177.0	837.7	15.5	14.5

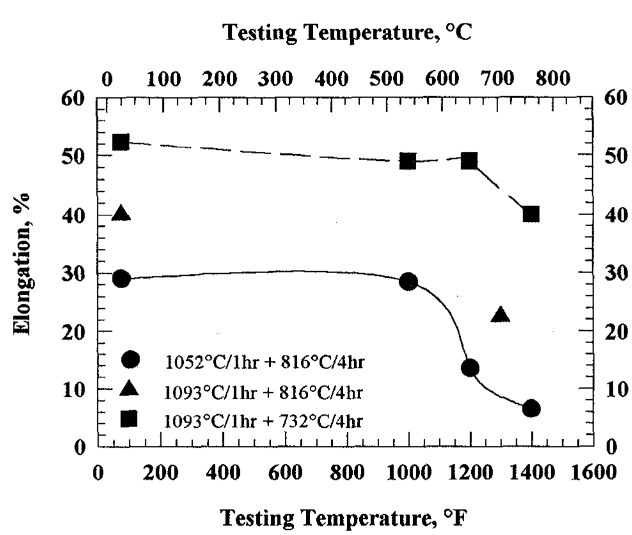

Figure 12. Tensile elongation values for AEREX™ 350.

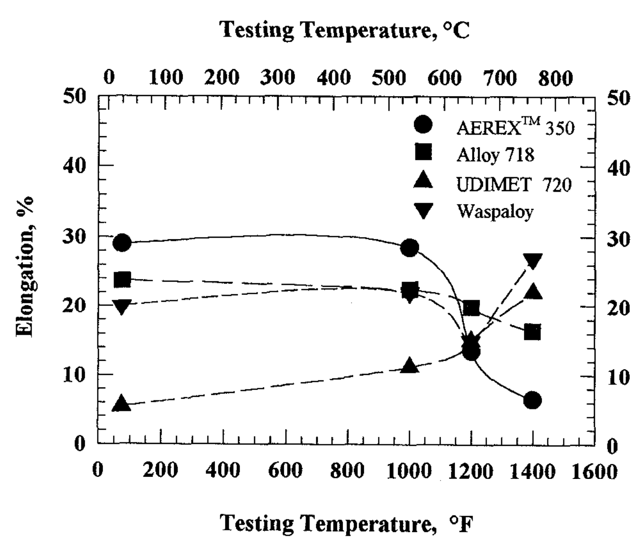

Figure 13. Tensile elongation values for selected superalloys.

Superalloys strengthened by γ′ (with sufficient titanium) are susceptible to η phase formation just as alloys with sufficient niobium and strengthened with γ″ may precipitate delta phase (as in alloy 718). Some studies have shown the presence of η phase to be deleterious to properties.[13,14] Degradation occurs when the η phase precipitates as a cellular structure or the volume fraction of η becomes too great in an overaged condition. Other studies have shown that η may enhance properties with the proper heat treatments to obtain an appropriate microstructure.[15,16] It has been demonstrated in this paper that the presence of η has an effect on both strength and ductility. The η in this alloy has been observed to have a Widmanstätten plate-like morphology. Therefore, the loss in ductility utilizing a subsolvus solution treatment temperature is not due to a cellular η precipitating at the grain boundaries.

Conclusions

The precipitating phases which occur in AEREX™ 350 include both γ′ and η. The presence of the η phase helps to provide high temperature strength to the alloy. It appears that the η phase precipitates as low as 788°C (1450°F) and the solvus temperature is between 1093°C (2000°F) and 1052°C (1925°F), whereas γ′ is present as low as 732°C (1350°F) and the solvus temperature is below 1038°C (1900°F).

The γ′ appears to be uniformly distributed in the matrix and coexists with η at temperatures above 788°C (1450°F). The η precipitates are generally plate shaped and appear predominately on the grain boundaries. Overaging temperatures, however, increase the apparent volume fraction of η, and η precipitates in the grain interiors on preferred crystallographic planes. The heat treatment is critical in determining the distribution and morphology of precipitating phases which, in turn, may be used for mechanical property optimization.

Work is in progress to further refine the heat treatment process parameters which will influence the final structure and properties of the alloy. It is anticipated that two heat treatments will be developed: one which will provide the best creep and stress rupture properties using a solution treatment above the η solvus temperature and one which will take advantage of the presence of η to provide elevated temperature strength. Long term, elevated temperature exposures will also be investigated to determine the effect on the microstructure.

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