HEAT TREATMENT VARIABLES ON MICROSTRUCTURE

AND MECHANICAL PROPERTIES OF CAST PWA 1472

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Abstract

Cast PWA 1472, a high strength modification of cast Alloy 718, was studied via cast test bars that were heat treated under a different conditions in order to relate variety of microstructure with mechanical properties, in particular stress rupture life at 1200°F and 90 ksi. Cooling rate from the solution temperature had the greatest effect by increasing life by more than 300 times from the fastest cooling rate of 40°F/min to the slowest cooling rate of 5°F/min. This slow cooling rate enhanced more strengthening phase precipitation, thereby increasing stress rupture performance. It produced a large amount of γ " and γ ', even without an aging heat treatment. Also, specimens excised from a commercial diffuser casing cast from the same heat as the test bars provided similar properties. In both cases, stress rupture test results, coupled with tensile test results at 1200°F, were at least 25 pct higher than cast 718 bars from the same source and confirm an earlier report on PWA 1472. The alloy exhibited residual segregation regardless of heat treatment and remnants of dendritic and cast nature. Because segregation leads to uneven precipitation, mechanical properties probably have not been maximized for large and fine grain castings.

Introduction

Investment castings of a wide variety of sizes and shapes are now being made of Alloy 718 for aircraft engines and this successful development has been described in the 1989 and 1991 Symposia. Also, as higher strength versions of cast Alloy 718, both Rene 220C and PWA 1472 were introduced in the 1989 Symposium. In his overview on the development of cast PWA 1472 (which was not published), Hatala⁽¹⁾ related that this was done by principally reducing the Cr content to 12 wt pct and raising Ti content to 2 wt pct and Nb content to 6 wt pct. Reducing the

> Superalloys 718, 625, 706 and Various Derivatives Edited by E.A. Loria The Minerals, Metals & Materials Society, 1994

Cr content to a level outside the current cast Alloy 718 specification requirement effectively suppressed the formation of Laves phase during solidification. From his presented data, Hatala classified PWA 1472 as 25 pct stronger than regular Alloy 718 and with creep properties at 50° F to 100° F higher temperature. Also, he showed a number of commercial castings that have been produced from this modified Alloy 718 including engine diffuser and turbine cases.

1991 Symposium, Radavich⁽²⁾ presented the initial At the metallographic study of PWA 1472 and he revealed an electrolytic etch preparation technique which can be used to characterize the phases found in samples which were given various thermal treatments. He noted that the lower Cr level is designed to decrease the segregation effect of Cr and to allow a higher Nb level without increasing the overall segregation behavior of the Since the Nb content determines the amount of alloy. strengthening phases, PWA 1472 should show higher strength levels after appropriate heat treatment. He concluded that the phases found in cast PWA 1472 are the same as those found in cast Alloy 718 and the degree of segregation during solidification depended upon the Nb content and the cooling rate.

As a continuation of the study, this paper relates the microstructure in cast test bars given various thermal treatment with mechanical properties, in particular stress rupture test

	PWA 14	472 Spec.	Heat
Element	Min.	Max.	715935
Nickel		Bal.	Bal.
Iron	17.00	19.00	18.00
Chromium	11.00	13.00	11.50
Niobium (+Ta)	5.75	6.25	6.20
Molybdenum	2.80	3.30	3.00
Titanium	1.75	2.25	2.00
Cobalt		1.00	0.05
Aluminum	0.40	0.80	0.46
Carbon	0.02	0.06	0.04
Manganese	-	0.35	0.030
Silicon	-	0.15	0.120
Copper	-	0.10	0.009
Zirconium	-	0.03	<0.01
Phosphorus	-	0.015	0.016
Sulfur	-	0.005	0.006
Boron	-	0.005	0.004
Lead	-	0.0005	<0.0002
Selenium	-	0.0003	<0.0001
Bismuth	-	0.00003	<0.00005
Oxygen	To Be	Reported	0.004
Nitrogen	To Be	Reported	0.004

Table I PWA 1472 Spec and Composition of Heat 715935 in Weight Percent

results. Included were stabilization heat treatment and solution temperature, cooling rate from solutioning and aging temperature in an effort to evaluate process development. Initial study of cast test bars screens the proper composition, heat treatment and level of properties but it is then necessary to study the full-scale casting in order to ascertain structure, properties and uniformity. Because the test bars were cast to size, the microstructure could be quite different than that in fine or coarse grain castings. Hence, the degree of segregation and the grain size requirement would determine the nature of the thermal treatment imposed on the casting prior to aging. The microstructure of coarse and fine grain castings is presented in the thesis by Wardell⁽³⁾ along with other aspects of this work that could not be included in the present paper.

Material and Procedure

The compositional range per PWA 1472 specification and the composition of Heat 715935 from which the test bars were cast to size are given in Table I. The processing and thermal treatments were provided by Hatala and associates at PWA⁽⁴⁾. All test bars received a HIP treatment at 2175°F for 4 hours at 15 ksi (103 MPa). This treatment closes porosity in the cast condition. Grain growth can occur at high temperatures and long treatment times required for successful HIPing. In this regard, Wardell⁽³⁾ revealed that the as cast microstructure was coarse grained when HIPed at 2175°F and fine grained when HIPed at 2050[°]F. Prior to the HIP treatment, the cast test bars were given the homogenization treatments listed in Table II. After the HIP treatment, some bars were stabilized at 1600°F and all were given the solution temperature and cooling rate listed in Table II and then aged either at 1350°F or 1400°F. A Taguchi heat treat development evaluation was used in setting up the conditions for each bar listed in Table II. It is apparent from the heat treatments that HIP closure of casting porosity and the homogenization of segregation were of prime interest as they would affect structure response and mechanical properties.

Heat <u>Treat</u>	<u>Stabilization</u>	Solution Temp.	Solution Cooling Rate	Age	
1	-	1850°F	40°F/Min.	1350°F (8) +	1225°F (8)
2	-	1925°F	5°F/Min.	11	11
3	1600°F (1)	1850°F	5°F/Min.	÷ t	11
4	1600°F (1)	1925°F	40°F/Min.	H	н
5	-	1850°F	5°F/Min.	1400°F (4) +	1225°F (2)
6	-	1925°F	40°F/Min.	H	H
7	1600°F (1)	1850°F	40°F/Min.	ET	11
8	$1600^{\circ}F(1)$	1925°F	5°F/Min.	11	11
$\tilde{9}^{(1)}$	1600°F (1)	1850°F	40°F/Min.	11	11
	Heat Tr	eatment: PHF + S	$HIT^{(1)} + HIP^{(2)} +$ Solution + Age	Stabilization	

Table II Heat treat Details on Cast Test Bars of PWA 1472



The structural response of the various specimens was evaluated by optical and SEM of electropolished and etched surfaces as described by Radavich⁽²⁾, as well as appropriate EDAX and x-ray diffraction, per Wardell⁽³⁾. Mechanical properties were determined on specimens machined from the cast bars, with tensile tests being performed at ambient and at 1200°F and stress rupture testing at 1200°F under 90 ksi (621 MPa) or 100 ksi (690 MPa). Also, for comparison purposes, PWA provided results on specimens machined from an actual casting produced from the same heat as the test bars.

Results

The 1200°F tensile test results are listed in Table III. There is some variation in the yield and ultimate strength and the ductility values which can be attributed, in part, to the differences between the individual thermal treatments listed in Table II. It is evident that ultimate strength and elongation are higher in bars B-2, B-3, B-5 and B-8 which were given the

> Table III Tensile Test and Stress Rupture Results on Cast Test Bars of PWA 1472

1200°F Tensile Properties

Spec.	Heat <u>Treatment</u>	0.2% YS (Ksi)	UTS <u>(Ksi)</u>	EL (Z)	RA (7.)
	1	1/0 6	157 3	4.7	16 0
B-1	1	140.0	121.2	4.5	10.9
B-2	2	142.9	160.7	8.1	13.3
B-3	3	143.2	166.6	6.6	11.0
B-4	4	146.0	155.9	6.0	23.4
B-5	5	144.9	163.4	4.7	14.0
B-6	6	150.3	155.3	4.1	24.1
B-7	7	145.1	154.2	4.6	22.7
B-8	8	138.2	161.9	6.3	12.5
B-0	0	147 8	157 8	53	19.9

1200°F/90 Ksi Stress Rupture Properties

Spec.	Heat <u>Treatmen</u>	Rupture Time (Hours)	EL (Z)	RA (<u>7)</u>
B-10	1	6.9	1.6	3.2
B-11	2	329.0	0.21	Disc.
B-12	3	298.0	1.6	2.8
B-13	4	2.2	2.1	5.5
B-14	5	308.2	0.21	Disc.
B-15	6	0.6	-	-
B-16	7	27.0	2.1	2.4
B-17	8	328.7	0.32	Disc.
B-18	9	89.1	1.5	2.5
Heat	Treatment:	PHHT ⁽¹⁾ + HIP ⁽²⁾	+ Stabili	ization

+ Solution + Age

Notes: (1) PHHT - Pre-HIP Homogenization Treatment 2075°F (4) + 2100°F (4) (2)^{used} on samples <u>except</u> No. 9 which received no PHHT. All HIP at 2175°F (4) - 15 ksi

slow cooling rate of $5^{\circ}F/min$ from the solution temperature of $1850^{\circ}F$ or $1925^{\circ}F$ and then aged at $1350^{\circ}F$ or $1400^{\circ}F$. There was very little microstructural difference in the bars that were solution heat treated at $1850^{\circ}F$ and $1925^{\circ}F$. If anything, there seemed to be more δ plates in the bars solution treated at the lower temperature. The effect of a $1950^{\circ}F$ solution treatment on bar B-3 was sufficient to resolution all phases except nitrides and carbides.

The stress rupture test results at $1200^{\circ}F$ under 90 ksi are also listed in Table III for the various heat treatments given in Table II. Here, there is more conclusive evidence of the beneficial effect of the slowest cooling rate from the solution temperature in increasing rupture life, as seen in bars B-11, B-12, B-14 and B-17. All of these bars, cooled at $5^{\circ}F/min$ from either $1850^{\circ}F$ or $1925^{\circ}F$, produced a rupture life that was at least 300 times longer than that exhibited in the bars that were cooled at a faster rate of $40^{\circ}F/min$ from either temperature.

Figures 1 and 2 each show two different bars given the same heat treatment. These bars were compared to determine whether or not the microstructure was consistent between them. As shown in Figure 1, when bars B-1 and B-10 were given the same heat treatment, the microstructures are very similar. Blocky carbides and δ plates are common to both bars. There is also a lack of smaller γ " and γ ' precipitation between the δ plates. As seen in Figure 2, bars B-2 and B-11 exhibit similar microstructures. Blocky carbides are surrounded by a few δ plates and heavy γ " and γ ' precipitation.

The cooling rate from the solutioning temperature, before the treatment, has the most significant effect aging on microstructure of all treatments studied. The stress rupture tests revealed a significant difference in the properties of those bars that were cooled at 40° F/min and those cooled at 5^{0} F/min. The slow cooled bars produced very long stress rupture lives. Figure 3 shows the difference in the microstructure of slow cooled and fast cooled bars. The slow cooled bars show profuse γ " and γ ' precipitation, while the fast cooled bars have much less and much finer precipitation. The fast cool appears to inhibit the nucleation of γ^{\dagger} and γ' . During the slow cool, the strengthening precipitates nucleate and then grow more quickly during the aging treatment.

The bars which were heat treated with and without the $1600^{\circ}F$ stabilization heat treatment following the HIP treatment showed more δ phase plates after the $1600^{\circ}F$ stabilization treatment. The $1600^{\circ}F$ heat treatment is in the temperature range for γ " phase and δ plate precipitation, but the following 1850 or $1925^{\circ}F$ solution heat treatments would be expected to resolution the γ " phase and most of the δ plates present. However, if the alloy is still sufficiently segregated, δ plates may persist to a higher solvus temperature. This segregation effect was evident as more δ plates were visible in the bars solutioned at $1850^{\circ}F$.

Two aging temperatures, 1400⁰F and 1350⁰F were studied in this program. The higher aging temperature resulted in larger precipitates. This was easier to see in the bars that were slow



Figure 1 Comparison of the microstructure of samples 1 and 10 at 2000X.

cooled, via bars B-12 and B-14 in Figure 4. Bar B-14 treated at 1400° F aging temperature had larger γ " precipitates than bar B-12. There was less difference in the bars that were cooled at the faster rate, and it was more difficult to detect a change in size.

The effect of 5° F/min slow cool with no aging treatment was examined in bar B-4 resolutioned at 1925°F for one hour and then slow cooled at 5° F/min to 1400° F. At low (100X) magnification the cast nature of the alloy was evident by the dendritic pattern and the small subgrain boundaries seen throughout the microstructure. At 2000X, γ " can be seen to have precipitated in the interdendritic areas. At 10,000X per Figure 5A, the γ " and possibly γ ' have already precipitated quite extensively in the interdendritic regions, and Figure 5B shows that there is also a fine γ " precipitate in the dendritic regions. This means that much of the γ " and possibly γ ' precipitation is occurring during cooling.



Figure 2 Comparison of samples 2 and 11 at 2000X.

Discussion

It is interesting to make a comparison of the mechanical property results on PWA 1472 with the results from the same source on cast + HIP bars of PWA 1469 (Alloy 718) presented recently by Schirra et al⁽⁵⁾ After appropriate heat treatment of cast bars of PWA 1469 having a low level of Laves phase, their 1200° F tensile test results were 95.7 ksi (662 MPa) yield and 115.9 ksi (725 MPa) ultimate strength, with 12 pct elongation. These values can be compared to the average values of 142.3 ksi (979 MPa) yield and 163.2 ksi (1124 MPa) ultimate strength, with 6.5 pct elongation, for PWA 1472 bars B-2, B-3, B-5 and B-8 which were slow cooled at 5° F/min from either 1850°F or 1925°F. Even if the average results for all nine bars are considered, irrespective of heat treat details, the average values of 134.8 ksi (928 MPa) and 148.8 ksi (1026 MPa) for PWA 1472 are significantly higher than those obtained on good quality PWA



Figure 3 The cooling rate from the solutioning temperature to the aging temperature has a great effect on the resulting microstructure. The slow cool (a) greatly enhances γ " precipitation over the fast cool (b). 10,000X

1469 (Alloy 718). Also, a comparison can be made on the smooth stress rupture test results at $1200^{\circ}F$ even when the above four PWA 1469 material was tested under 80 ksi (552 MPa) whereas the above four PWA 1472 bars were tested under 90 ksi (620 MPa). The rupture life was 104 hours with 7.5 pct elongation for the former and beyond 300 hours without rupture in bars B-11, B-14 and B-18. Rupture occurred in 298 hours in bar B-12. However, it is recognized that the three remaining bars, with different heat treatments, failed in very short times for some reason. Thus, under the optimum thermal conditions studied, PWA 1469).



Figure 4 Comparison of the γ " precipitation resulting from a (a)1400°F aging temperature versus a (b)1350°F aging temperature. 2000X

The thermal conditions and the resulting grain size in casting test bars are different than those occurring in a commercial casting. However, when obtained from the same heat (715935), it is noteworthy to make the following comparison with specimens excised from a gas turbine engine diffuser casing, using a grain refining process. The results listed in Table IV reveal that the 1200° F tensile properties are at least equal to the best results obtained on test bars when the same HIP conditions are employed (2175° F/15 ksi). Also, the smooth stress rupture life is comparable since the casting specimens were tested at a higher, 105 ksi (724 MPa) stress at 1200° F instead of the 90 ksi (620 MPa) stress at 1200° F imposed on the test bars. As shown in the Schirra et. al.⁽⁵⁾ paper on PWA 1469 (Alloy 718), these



Figure 5 Precipitation of γ " and γ ' in the (a) interdendritic regions and (b) dendritic regions. 10,000X

initial results confirm the necessity of reducing Cr content and using a pre-HIP homogenization heat treatment in conjunction with a $2175^{\circ}F$ HIP cycle to eliminate any Laves phase without melting it.

Because of space restrictions, the reader is referred to the Wardell thesis⁽³⁾ for other results of the study. It should be noted that specimens were given a subsequent TAG treatment of one hour at 1600°F in order to determine the amount of residual segregation. Results showed that the Nb content was at least 4 pct. throughout. However, since the alloy contains 6 pct Nb, residual segregation can exist which probably caused sporadic Table IV Tensile Test and Stress Rupture Results on Specimens Cut from Production Casting of PWA 1472

Room Temp. Tensile Properties UTS EL RA 0.2% YS (%) (%) (Ksi) (Ksi) Specimen $C^{-1}_{C^{-2}(2)}^{(1)}$ 3.5 4.8 163.0 191.3 4.0 7.8 179.8 200.2 1200°F Tensile Properties 0.2% YS UTS EL RA Specimen (Ksi) (Ksi) (%) (%) C-3(1) 136.5 158.1 4.4 7.1 155.4 167.9 4.0 10.4

1200°F/105 Ksi Stress Rupture Properties

Specimen	Rupture (Hours)	EL (Z)	RA (%)	
C-5(1) C-6(2)	117.1 196.5	2.3 3.1	6.0 5.4	
Specimen Source: Heat Treat:	Production I HIP* + 1600° + 1400°F (4)))) + 1225	Case 1850°F °F (2)	(1)* +

Notes: ⁽¹⁾_{HIP} @ 2050 °F (3) - 15 ksi ⁽²⁾_{HIP} @ 2175°F (4) - 15 ksi

mechanical behavior. Delta plates were present and all specimens studied showed some degree of residual segregation regardless of heat treatment. Because segregation leads to uneven precipitation, mechanical properties probably have not been maximized for large and fine grain castings.

Some recommendations from the results of this study can be made:

1. Further testing after longer solutioning times should be undertaken to determine the effect of reduced segregation on mechanical properties. Economic factors related to longer solution heat treatments at higher temperatures should be considered in determining the final heat treatment for this alloy.

2. Further testing of fast cooled samples with longer aging times should be performed to determine whether the same degree of precipitation and mechanical property enhancement can be achieved as with slow cooled samples.

<u>Conclusions</u>

Cast + HIP test bars of PWA 1472 were heat treated under a variety of different conditions in order to relate microstructure with mechanical properties, in particular rupture life at 1200° F and 90 ksi.

The stress rupture tests showed that the cooling rate had the most significant effect on life. Specimens cooled at 40° F/min failed in a very short time, but cooling at a rate of 5° F/min increased stress rupture life by more than 300X.

Resolutioning and performing a slow cool of $5^{\circ}F/min$ showed that the γ " and possibly γ ' precipitated to a great extent before the start of the aging steps.

Solution temperature, stabilization, and age temperature variables produced much less dramatic results in the stress rupture life.

The microstructure resulting from the two cooling rates was very different. A slow cool produced much γ ", the major strengthening phase. While a fast cool resulted in almost no γ " precipitation.

The alloy still exhibited signs of Nb segregation. Segregation causes uneven precipitation in the alloy and most likely decreases the mechanical properties of the material or causes sporadic behavior. Delta plates were present and all specimens studied showed some signs of inhomogeneity (remnants of dendritic and cast nature).

Acknowledgements

The authors thank Pratt & Whittney Aircraft for providing the materials and their mechanical properties via Robert W. Hatala, and Niobium Products Company for the financial support of this study.

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