COMPARISON OF γ'/γ'' PRECIPITATES AND MECHANICAL PROPERTIES IN MODIFIED 718 ALLOYS

Encai Guo, Fengqin Xu and E.A. Loria

Central Iron and Steel Research Institute
Beijing, China

and

Niobium Products Company
Pittsburgh, PA, USA

Abstract

Microstructural instability and loss in strength of Alloy 718 at and above 650°C is caused by the dissolution of the precipitation strengthening γ'' phase and the formation of the brittle and crack-like δ phase. Two improved compositional modifications of Alloy 718 were given heat treatments that could change the morphology of the coexisting γ'/γ'' precipitates. A conventional non-compact γ'/γ'' structure was obtained in one alloy and a compact γ'/γ'' structure in the other alloy. A third alloy specifically designed to produce the compact γ'/γ'' cuboidal precipitate was included for comparison purposes. Higher tensile strength averaging 105 MPa (2.3 ksi) between 400 and 700°C (750 and 1300°F) and almost three times longer rupture life at 650°C (1200°F) under a stress of 686 MPa (99.5 ksi) were obtained with the non-compact γ'/γ'' precipitate compared to the compact γ'/γ'' precipitates and the non compact γ'/γ'' structure of conventional heat treated 718. Also obtained were significant improvements in tensile yield strength and stress rupture life at 483 to 724 MPa (70 to 105 ksi) in the 650 to 730°C (1350°F) temperature regime corresponding to γ'' instability in conventional 718. For this particular high stress range, a 100 hr rupture life at 25 to 40°F higher temperature was calculated for the modified alloy with the non-compact γ'/γ'' structure compared to the trendline established for today's high-quality 718. For this particular composition, the mechanical properties were apparently optimized by the size and spacing of the non-compact γ'/γ'' precipitates being more effective in impeding the motion of dislocations through the lattice. There was an increase in the primary strengthening γ'' phase, and the matrix and $\gamma' + \gamma''$ phases were strengthened by a tungsten addition to this alloy. Also, the grain boundary was strengthened by a small chain of M_6C phase which inhibited long range grain boundary sliding during stress rupture testing.

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Introduction

The goal of our study¹ has been to identify the alloying parameters that determine the strengthening precipitation phases in Alloy 718 and thereby increase the maximum operating temperature. This appears possible through minor changes in alloy composition and heat treatment.^{2,3} Our introductory paper⁴ revealed the results of different heat treatments than the conventional 718 heat treatment for two promising compositional modifications of 718 from our initial investigation¹ which showed superior properties when given the standard 718 heat treatment. The selected heat treatments produced a conventional non-compact mixture of γ'/γ'' particles in one alloy and a compact γ'/γ'' precipitate structure in the other alloy. For comparison purposes, Pineau's preferred composition⁵ that produces the compact γ'/γ'' cuboidal precipitate morphology with the appropriate heat treatment was included because it has been reported to provide better thermal stability above 650°C. Our aim was to evaluate the effect of these heat treatments on precipitate structure and mechanical properties. Particular emphasis was placed on the 650 to 730°C temperature regime corresponding to γ'' instability in conventional 718.

Materials and Procedure

The modifications of Alloy 718 were vacuum induction melted and cast as 23 kg ingots in the case of Alloys 5 and 7 and 5 kg ingot in the case of Alloy 3. After homogenizing for 24 hours at 1100°C and for 1 hour at 1160°C to minimize segregation effects, the ingots were hot forged into 32 mm bars. Specimens cut from the bars provided the compositions listed in Table I. As selected from the introductory paper,⁴ Alloy 5 was solution treated at 1030°C for 1 hr, air cool, 800°C

Table I Chemical Composition of Alloys									wt. %
Alloy	C	Cr	Mo	Ti	Al	Nb	Fe	W	B
3	0.056	17.58	2.85	0.97	0.86	5.51	17.00		0.0033
5	0.048	16.60	3.09	0.98	0.93	5.57	13.71	2.30	0.0019
7	0.059	17.20	2.98	1.20	1.19	4.95	19.23		0.0041
Alloy	Ti	Al	Nb	Al+Ti+Nb	Al/Ti	Al+Ti/Nb	at. %		
3	1.18	1.85	3.44	6.47	1.57	0.88			
5	1.21	2.04	3.55	6.80	1.69	0.92			
7	1.48	2.33	3.16	6.97	1.57	1.21			

for 1.5 hr, then furnace cool (50°C/hr) to 650°C, hold for 16 hr and air cool. Alloy 7 and Alloy 3 were solution treated at 980°C for 1 hr, air cool, 850°C for 1 hr, then furnace cool (50°C/hr) to 650°C, hold for 16 hr and air cool. These heat treatments produced a γ matrix grain size of ASTM5-6 in Alloys 5 and 3 and ASTM6-7 in Alloy 7. The metallography, tensile, impact and stress rupture testing was done on the heat treated bar stock following the standard procedure in each case.

Results

Transmission electron micrographs, per Figure 1, revealed that the heat treatment on Alloy 5 produced a conventional mixture of thin disk-shaped γ'' and small round γ' particles, with a significant number of the γ'' bound to γ' particles.

Figure 1. Transmission electronmicrographs of Alloy 5 after specified heat treatment. (a) Dark field, X80000. (b) Bright field, X80000. (c) Dark field, X115000.

Figure 2. Transmission electronmicrographs of Alloy 7 after specified heat treatment. (a) Dark field, X80000. (b) Dark field, X105000.

Basically, it was not the γ'/γ'' compact precipitate morphology. The γ'' particles were thinner and spaced closer together than observed in Alloys 3 and 7. The γ' diameter was about $0.018\ \mu\text{m}$ and the γ'' length about $0.025\ \mu\text{m}$ in Alloy 5. There was some evidence of M_6C phase distributed as a small chain in part of a grain boundary but no needle δ phase was observed in the grain boundaries or within the grains. The heat treatment employed on Alloy 7 produced the distinctive compact γ'/γ'' precipitate morphology shown in Figure 2. The γ' diameter was about $0.033\ \mu\text{m}$ and the γ'' length was about $0.023\ \mu\text{m}$. A very small amount of δ phase was observed in the grain boundary but none was seen intragrain. The heat

Figure 3. Transmission electronmicrographs of Alloy 3 after specified heat treatment. (a) Dark field, X80000. (b) Bright field, X80000.

treatment employed on Alloy 3 also produced the compact γ'/γ'' precipitate shown in Figure 3. The γ' diameter and γ'' length both measured $0.038\ \mu\text{m}$ and the distribution of δ phase in Alloy 3 was similar to Alloy 7. Generally, for the best creep strength, γ' particles should be very small but of an optimum size to achieve a good combination of strength and ductility. In this regard, the γ' particles in Alloy 5 are 50 pct smaller than the γ' particles in Alloys 7 and 3.

Chemical analyses of the extraction phases from Alloys 5 and 7 are listed in Table II. In addition, Alloy 5 has 0.58 wt.pct (NbC + TiC) and Alloy 7 has 0.42 wt.pct (NbC + TiC). The analytical results reveal no obvious difference in the total amount of $\gamma' + \gamma''$ extracted from each material. The amounts of Ti and Al which

Alloy	Ti	Fe	Mo	Cr	Ni	W	Nb	Al	Total
5	0.81	0.33	0.12	0.32	14.25	0.62	3.45	0.78	20.68
7	0.99	0.40	0.05	0.24	15.67		2.25	0.87	21.47

are the main elements forming γ' phase are higher in Alloy 7 which agrees with the large cube-shaped γ' particles observed in the transmission electron micrographs. Also, the calculated ratio of the volume fraction of γ' over volume fraction of γ'' phase from dark field TEMs is roughly about 4 for Alloy 7 compared to about 2 for Alloy 5. Conversely, the amount of γ'' phase, which is the primary strengthening phase in 718 type compositions, is obviously higher in Alloy 5. Strictly on the basis of the compositional ratio of Nb/(Al+Ti+Nb) in at.pct, the values are 52.2 pct for Alloy 5 and 45.3 pct in Alloy 7. Finally, in Alloy 5, the $\gamma' + \gamma''$ is strengthened by the 0.62 wt.pct W which is about 30 wt.pct of the added amount (plus 0.12 wt.pct Mo vs. 0.05 wt.pct Mo in Alloy 7) with the remainder providing solution strengthening of the γ matrix.

The mechanical properties of the three alloys are listed in Tables III to V.

Table III Mechanical Properties of Alloy 5 after Specified Heat Treatment												
Tensile Test Results							Stress Rupture Test Results					
Temp °C	0.2% MPa	Yield ksi	Ultimate MPa	ksi	El %	RA %	Temp °C	Stress MPa	ksi	Time hr	El %	RA %
25	1120	162	1460	212	21.0	33.7	650	686	99.5	273.2	10.5	25.4
25	1110	161	1485	215	22.3	36.6	650	686	99.5	394.0	13.5	29.7
400	1085	156	1565	227	21.5	32.5	680	724	105	28.0	13.4	32.4
400	1065	154	1505	218	20.2	34.7	680	724	105	34.9	14.2	19.7
650	1005	146	1275	185	23.1	30.4	700	638	92.5	30.4	6.4	11.2
650	1040	151	1320	191	27.5	33.3	700	638	92.5	52.9	10.8	19.0
700	925	134	1140	165	9.9	16.1	700	638	92.5	31.8	8.2	18.3
730	890	129	1060	154	11.4	9.7	730	483	70	63.6	13.3	33.8
730	900	131	1055	153	9.5	11.2	730	483	70	59.4	17.4	40.4

Table IV Mechanical Properties of Alloy 7 after Specified Heat Treatment												
Tensile Test Results							Stress Rupture Test Results					
Temp °C	0.2% MPa	Yield ksi	Ultimate MPa	ksi	El %	RA %	Temp °C	Stress MPa	ksi	Time hr	El %	RA %
25	1040	151	1490	216	19.0	34.1	650	686	99.5	180.8	8.8	20.1
25	1045	152	1505	218	19.3	34.0	650	686	99.5	61.1	2.9	4.0
							650	686	99.5	180.3	12.9	20.0
400	1030	149	1400	203	16.0	31.4	680	724	105	11.2	7.7	10.9
400	995	144	1375	199	16.1	33.0	680	724	105	11.8	9.3	17.2
650	985	143	1225	178	22.4	39.5	700	638	92.5	16.7	15.9	26.4
650	980	142	1220	177	24.6	47.0	700	638	92.5	23.1	15.2	24.7
700	870	126	1075	156	13.4	15.7	700	638	92.5	22.9	19.1	24.7
700	920	133	1070	155	13.6	17.6	730	483	70	13.1	10.7	21.9
730	825	120	940	136	12.0	13.9	730	483	70	22.9	24.6	39.4
730	825	120	965	140	11.0	16.1	730	483	70	26.1	10.4	18.7

Table V Mechanical Properties of Alloy 3 after Specified Heat Treatment												
Tensile Test Results							Stress Rupture Test Results					
Temp °C	0.2% MPa	Yield ksi	Ultimate MPa	ksi	El %	RA %	Temp °C	Stress MPa	ksi	Time hr	El %	RA %
25	1110	161	1490	216	18.5	33.0	650	686	99.5	173.5	10.4	14.6
25	1150	167	1535	223	17.5	31.4	650	686	99.5	107.0	36.6	40.8
400	1030	149	1405	204	14.9	32.8	700	638	92.5	9.5	19.3	43.5
650	985	143	1195	173	30.9	53.2	700	638	92.5	13.9	22.6	44.6
650	985	143	1210	176	29.7	43.7	700	638	92.5	23.3	10.6	14.6
700	905	132	1035	150	25.2	35.0	730	483	70.0	28.1	18.5	39.2
730	850	123	940	136	21.0	26.3	730	483	70.0	29.9	25.1	38.9

Figure 4. Variation in tensile strength and 0.2 pct yield strength of Alloys 3, 5, 7 and 718 with increasing temperature up to 730°C.

Depicted graphically in Figure 4, the tensile strengths at room temperature are nearly identical but there is a difference in the yield strength values which are below the value for Alloy 718. However, at test temperatures between 400°C and 650°C, the strength properties of Alloy 5 are significantly higher, and the increase in tensile strength is attributed to the high work hardening rate during plastic deformation. Then, the improvement continues over the expected decline that

Figure 5. Variation in impact strength of Alloy 5 and Alloy 7 between ambient and 700°C.

occurs above 650°C. A tensile strength of 1140 MPa (165.3 ksi) and yield strength of 925 MPa (134.2 ksi) at 700°C and 1058 MPa (153.1 ksi) and 897 MPa (130.1 ksi) at 730°C for Alloy 5 are noteworthy. On the other hand, the values for Alloys 7 and 3 are significantly lower and nearly identical throughout the temperature range and closely similar to Alloy 718 up to 700°C. As seen in Tables III to V, the corresponding elongation values are clustered around 20 pct at room temperature and increase generally at 650°C but then decrease in a range of 10 to 16 pct at 730°C. Reduction of area follows a similar trend and both are considered satisfactory.

From a large number of low cycle fatigue and fatigue crack propagation experiments on Alloy 718, Xie⁶ has concluded that LCF decreases with increasing temperature mainly because of the decrease in tensile strength and ductility. On this basis, the fact that Alloy 5 produced superior tensile strength and ductility would indicate that LCF life would be improved. Finally, Figure 5 shows the impact test results between ambient and 700°C and the higher values obtained on Alloy 5 compared to Alloy 7.

Stress rupture data are recorded in Tables III to V and the average life of each alloy at temperatures between 650°C and 730°C is compared in the bar graphs of Figure 6. At the present peak temperature of 650°C for today's 718 under a stress of 686 MPa (99.5 ksi), Alloy 5 lasts an average of 348 hr which is three times the 116 hr life obtained on Alloy 718. Practically the same degree of improvement occurs over Alloys 7 and 3 which had the same rupture life of 140 hr. Also, the superiority of Alloy 5 continues in the other three comparisons. At 700°C under a stress

Figure 6. Comparison of stress rupture times obtained on Alloys 3, 5, and 7 for the specified temperatures and stresses, and the respective elongation and reduction of area values.

of 638 MPa (92.5 ksi) and at 730°C under a stress of 483 MPa (70 ksi), Alloy 5 had two or three times the rupture life of Alloys 7 and 3. The corresponding elongation and reduction of area values were satisfactory in all cases and revealed no embrittlement tendency over the 650 to 730°C range. The phase stability of Alloy 5 is confirmed by these results of long survival at these high temperatures and stresses whereas conventional Alloy 718 inherently suffers significant deterioration in properties under these test conditions.

In examining the microstructures of specimens after stress rupture testing (and also impact testing), it was recognized that there would be internal stresses, dislocations and slip bands in the TEM foils. The very close spacing of the thin γ'' plates continued in Alloy 5, per Figure 7, after it ruptured in 394 hours at 650°C

Figure 7. Transmission electronmicrographs of Alloy 5 after rupture in 394 hours at 650°C and 683 MPa (99.5 ksi).
(a) Bright field, X10000. (b) Bright field, X80000.
(c) Dark field, X80000.

Figure 8. Transmission electronmicrographs of Alloy 5 after rupture in 63.4 hours at 730°C and 483 MPa (70 ksi).
(a) Bright field, X10000. (b) Bright field, X80000.
(c) Dark field, X115000.

Figure 9. Transmission electronmicrographs of Alloy 7 after rupture in 22.9 hours at 730°C and 483 MPa (70 ksi). (a) Bright field, X17000. (b) Bright field, X60000. (c) Dark field, X60000.

under a stress of 686 MPa (99.5 ksi). A tendency for γ'' phase growth was seen in some areas but with no change in other areas. Aside from the M_6C phase, there was no δ phase in the grain boundaries and none was seen within the grains. The microstructure of Alloy 5 after surviving 63.6 hours at 730°C under 483 MPa (70 ksi), per Figure 8, revealed slight tendency of γ'' growth but the form and amount of M_6C in the grain boundaries did not change and there was no δ phase anywhere. The microstructure of Alloy 7 after 22.9 hours at 730°C under 483 MPa (70 ksi) is shown in Figure 9. The compact γ'/γ'' precipitate showed no growth (coarsening) tendency. A small amount of δ phase was observed in the grain boundaries but there was no δ phase within the grains.

In these comparisons between Alloy 5 and Alloy 7, the improvement in rupture life of Alloy 5 can be attributed to the observed differences in the respective γ'/γ'' precipitates plus the effect of the tungsten addition in strengthening both the γ matrix and the $\gamma' + \gamma''$ phases. In addition, the formation of a small chain-like M_6C precipitate in Alloy 5 strengthened the grain boundaries and thereby contributed to the improvement in rupture life. These structural factors are controlled by the precipitate chemistry via optimum alloying additions (and appropriate heat treatment). The second factor to enhance high temperature properties is a high precipitate content. In this respect, the higher amount of the primary strengthening γ'' phase in Alloy 5, the smaller size of the γ' precipitate, and the higher volume fraction of γ'' from the extraction data, as well as Nb percentage relative to total hardening element content are the contributing factors.

Discussion

The goal of our research has been to extend not only the service range to higher temperatures but also to improve properties at the present ceiling temperature of about 650°C for conventional 718. This upper limit has been related to microstructural instability caused by the formation of the brittle and

crack-like δ phase, and the accompanying dissolution of the precipitation strengthening metastable γ'' phase. Noteworthy is the higher tensile strength averaging 105 MPa (2.2 ksi) between 400 and 700°C and the almost three times longer stress rupture life of Alloy 5 compared to Alloy 718 at 650°C under a stress of 686 MPa (99.5 ksi). In age hardenable alloys, the heat treatment parameters determine mechanical properties and ultimately maximum service temperature. For the specified heat treatment, the mechanical properties of Alloy 5 were apparently optimized by the size and spacing of the γ'/γ'' precipitates being more effective in impeding the motion of dislocations through the lattice. When the comparison is made with the cuboidal γ'/γ'' morphology in Alloys 7 and 3, the coarsening rate of the γ'' phase growing in contact with the γ' precipitate and apart from it must have been lower. Although actual measurements are needed, it appears that the size and spacing of the precipitates in Alloy 5 provide maximum strengthening. In the initial study,¹ employing the standard 718 heat treatment, Alloy 5 had the shortest γ'' length and less wt.pct δ phase after 534 hours at 730°C of the five modified 718 alloys.

Tensile properties after long time service are more important than hardness because turbine disk burst (the designer's primary concern) is by overspeed wherein the ultimate strength is approached by the average tangential stress.⁷ Ductile failure occurs when the average tangential stress reaches a large fraction of the ultimate stress; tests show values below 0.9. The higher tensile strength of Alloy 5 compared to Alloy 718 (as well as Alloys 3 and 7) over the 20 to 730°C range is noteworthy, and it should be ascertained if these properties are maintained after long time exposure in the 650 to 730°C range. Pertinent, in this regard, is the prior result on Alloy 5, when given the standard 718 heat treatment, which showed no reduction but rather higher tensile strength after 534 hours at 730°C (1420 MPa). The study by Barker et al.⁸ of twenty years ago provides a comparison based on a criteria of tensile properties after thermal exposure. They concluded that Alloy 718 will not have large losses in strength until there is significant overaging of the γ' and γ'' precipitates and formation of large quantities of δ phase. This did not occur in 200 hr at 700°C (1300°F) but did occur in 800 hr at 700°C.

The introductory paper⁴ showed the conversion of the data on our five modified alloys, when given the standard 718 heat treatment, to 100 hr life via the Larson-Miller parameter and compared to the trend-line established for today's high-quality 718. The addition of the present data on Alloys 3, 5 and 7 to the prior data on Alloys 3 and 5 produced the composite diagram, Figure 10. In the high stress range, it is evident that Alloy 5 has a 100 hr rupture life at 25 to 40°F higher temperature than Alloy 718 whether the standard 718 or the modified heat treatment is employed. Although there are more data for the modified heat treatments than for the standard 718 heat treatment, it appears that there is no apparent difference between them and the precipitate structure in which only part of the γ'' is bound to the γ' particles in Alloy 5 provides superior results. Surprisingly, the compact γ'/γ'' precipitate morphology in Alloy 7 provides, at best, equivalent results to conventional 718 but, more often, inferior results throughout the stress range. And these results were also confirmed when Alloy 3 was given the modified heat treatment which provided essentially the same compact γ'/γ'' structure.

The subject alloys represent three ways that the composition of Alloy 718 can be modified to provide higher (Al+Ti)/Nb and Al/Ti ratios with higher

Figure 10. Conversion of stress rupture data for Alloys 3, 5 and 7 to 100 hour rupture life via the Larson-Miller parameter ($C=22$) compared to trend-line established for today's quality Alloy 718.

(Al+Ti+Nb) total hardener content. Although Alloy 7 possessed the highest values in each case, Alloy 5 provided better thermal stability at and above 650°C. In the manipulation of these elements, this result reveals that the correct balance of these elements is necessary in order to attain the optimum γ'/γ'' precipitate structure from a particular heat treatment. In this comparison, Alloy 5 had a higher Nb percentage with respect to total hardener element content which would provide a higher Nb percentage in the γ'' precipitate which would be expected to increase the coherency strains. As a consequence, higher alloy strength was attained with satisfactory rupture life, indicative of phase stability. Also, it should be recognized that Alloy 5 contained a W addition which would change the disposition of refractory elements in each phase, thereby modifying lattice mismatches between γ/γ' , γ'/γ'' and γ/γ'' . Finally, it should be acknowledged that our results pertain to bar stock processed from small VIM heats rather than the commercial VIM-VAR plus TMP for disk production which could provide an improvement. Non-uniform dispersion of the precipitate phases would be detrimental to mechanical properties but the fact that all of the results are closely similar would negate this possibility.

Conclusions

Superior mechanical properties were obtained with a non-compact γ'/γ'' precipitate produced by a specified heat treatment in a modified Alloy 718 compared to two other compositional modifications having a cube-shaped, compact γ'/γ'' precipitate with appropriate heat treatment. Higher tensile, impact

and stress rupture properties were measured up to 730°C, as well as in comparisons with conventional 718. The structural factors were controlled by the precipitate chemistry via optimum alloying additions and appropriate heat treatment. There was an increase in the primary strengthening γ'' phase and the γ' particles were smaller in this alloy. Both the matrix and the $\gamma' + \gamma''$ phases were strengthened by a tungsten addition. Also, the grain boundary was strengthened by a small chain of M_6C phase which inhibited long range grain boundary sliding during stress rupture testing.

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