EFFECT OF COMPOSITION ON SEGREGATION MICROSTRUCTURES AND MECHANICAL PROPERTIES OF CAST ALLOY 718

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Abstract

Variations in Nb, Cr, and Fe percent composition of alloy 718 are found to affect segregation, time and temperature of phase precipiation, and ultimately, the mechanical properties of cast alloy 718. Since the Nb participates in all phase precipitation of alloy 718, the Nb content determines the microstructural response for optimum precipitation and mechanical properties. When the Nb content is less than 4 wt%, such as what occurs in whitespots or the dendritic areas of cast alloy 718, the reactions which occur at lower temperatures need longer times than those used for heat treating conventional 718.

Room temperature tensile and 1200°F/90 KSI stress rupture tests were run on a modified (4 Nb - 14 Cr - 14 Fe) cast alloy 718 and standard cast 718 compositions. When both alloys were given a standard aging treatment of 1400°F/5 hours, the modified alloy did not meet the stress rupture life or yield strength. However, after an age of 20 hours at 1200°F, the modified alloy met the required tensile and stress-rupture properties. The mechanical properties are correlated to the microstructure.

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Introduction

The use of 718 in the cast form presents some interesting challenges in homogenizing and heat treat optimization. The sluggish response of the alloy to time and temperature, while an advantage for alloy stabilization, is a disadvantage when trying to diffuse elements for homogenization. The result of incomplete homogenization results in a composite-like alloy with regions of Nb content ranging from 10% in the interdendritic regions to close to 0% in the matrix. Radavich and Tien documented phase precipitation response to time and temperature for low Nb modifications of alloy 718. They concluded that greater than 4% Nb was necessary to precipitate γ'' at the conventional aging temperatures, and a lower temperature was necessary to precipitate γ'' at a less than 4% Nb 718 composition. In addition, lower Nb content presented a more homogenous as-cast alloy forming smaller amounts of Laves phase. While the low Nb alloy showed favorable structures there were no mechanical properties evaluated or processing optimization attempted. This study attempts to correlate, through mechanical properties, the relationship between the chemistry and phase precipitation of alloy 718.

Procedure

The study was divided into two phases. Phase I, evaluates the effect of Cr, Fe, and Nb variation on microstructure and homogenization in the as-cast and HIP (Hot Isostatic Press) form. Phase II focuses on the mechanical properties of a modified 718 composition 14% Cr, 14% Fe and 4% Nb as compared to conventional 718.

Phase I

Material Background. The material evaluated in Phase I was cast by Howmet Turbine Component Corporation (HTCC) from eight 15 lb VIM (Vacuum Induction Melt) heats of modified 718. The ingots were cast into circular double-gated test bar molds of varying diameters.

The nominal Fe, Cr, Nb composition of the eight modified heats are presented in Table I. The remaining elements were held within standard alloy 718 specifications. Heat #49 is within conventional 718 specifications while compositions of the remaining seven heats vary from the nominal composition specification.

Table I. Modified Cast Alloy 718 Segregation and DTA Data

XRD** Rating	Alloy Number	Cr	Fe	Nb	Ni	Solidification Peak--°F* Heat Up Solidus	Heat Up Liquidus	Cool Down Solidus	Cool Down Liquidus	Secondary phases cool down--°F*
7	46	13.94	19.67	4.02	57.73	2318	2503	2228	2453	Absent
8 least	47	13.74	13.70	3.94	63.43	2336	2516	2196	2435	Absent
5	48	13.95	13.65	5.30	62.46	2275	2491	2147	2437	Absent
1 most	49	18.06	19.40	5.50	51.90	2239	2464	2124	2399	2075-2123
3	50	14.09	19.70	5.35	56.22	2239	2489	2107	2415	2010-2041
2	51	17.90	14.0	5.38	58.08	2242	2473	2129	2413	Absent
6	52	17.90	13.99	4.03	59.44	2300	2494	2160	2444	Absent
4	53	18.11	19.90	4.10	53.25	2255	2498	2120	2440	1927-2008

*HTC - Howmet Turbine Components
**X-Ray Diffraction (10)

Heat Treatments. A 1 inch section of each of the eight bars was retained for as-cast microstructural evaluation. To simulate the thermal portion of the HIP cycle a second one inch section of each of the eight bars was subjected to a 2125°F/4 hour thermal treatment.

For homogenization evaluation, the thermal HIP samples were subjected to a short TAG heat treatment of 1600°F for one hour. This treatment precipitates the available Nb as γ'' in areas where the Nb content is 4% or higher which show up white by Secondary Electron Imagery. The TAGing of the γ'' precipitate is used as a visual indication of segregation only and not as a heat treatment for mechanical property response.

Phase II

Material Background. Two new VIM heats of modified alloy 718 were cast by Howmet into oversized test bars of the following chemistry:

	Nb	Cr	Fe	Ni	Mo	Al	Ti	Co	Cu	Si
#79	5.3	18.14	20.87	51.33	3.02	0.46	0.88	<.10	<.03	<.05
#78	4.17	14.39	16.32	60.84	3.17	0.40	0.79	<.10	<.03	<.05

Mechanical Testing. The test bars were subjected to a thermal cycle as specified by General Electric, for optimum room temperature tensile and 1200°F stress rupture properties. The total thermal treatments are as follows:

HIP	2125°F/15 KSI for 3-4 hours (GFC)
HOMOGENIZE	2000°F/1hour
SOLUTION	1750°F/1 hour
AGE	1400°F/5 hours-->1200°F/1 hour

Test bars of alloy #78 were subjected to three thermal treatments in addition to the above treatment to evaluate the mechanical properties and microstructural response of the low Nb, Cr, Fe alloy. The first bar was subjected to a solution and age at 1650°F/4 hours + 1400°F/20 hours, the second bar a two step age at 1400°F/20 hours + 1200°F/20 hours and the third bar a single age at 1200°F/20 hours.

Mechanical property tests were conducted according to ASTM standards. Tensile tests were conducted at room temperature at a strain rate of 0.050 inch per inch per minute to the 0.2 percent yield point and at 0.080 inch per inch per minute beyond the yield. Stress rupture tests were conducted at 1200°F and 90 KSI.

Microstructural Preparation. The samples of Phase I and Phases II were prepared the same way. Each sample was polished through 600 grit papers and electropolished for approximately 10 seconds at 25 volts, using an electrolyte solution of 20% H_2SO_4 10% HCl and 70% methanol. Each sample was electroetched in a CrO_3 etchant (172 cc H_3PO_4, 10 cc H_2SO_4, 16 g CrO_3) for 5-8 seconds at 5 volts.

Results and Discussion

Preliminary Investigation of Alloy 718 Modifications

Preliminary structural investigation of the eight modified heats of alloy 718 showed a correlation of degree of segregation with compositional values. The evaluation was based on qualitative optical microscopy and confirmed by x-ray diffraction of extracted residues. Results are listed in Table I as a ranking of most segregated sample (1) to least segregated sample (8). These preliminary findings indicate the most segregated alloy (#49) was high in Nb, Cr, and Fe content while the least segregated sample (#47) was low in Cr, Fe and Nb.

Howmet determined, by DTA, the effect of composition on the solidus and liquidus temperatures. A multiple linear regression equation illustrates the contribution of Nb, Cr, Fe and Ni to the solidus temperature as follows:

$$\text{solidus }(^\circ F) = 5325.242 - 35.29837\cdot(\text{wt\% Cr}) - 31.9217\cdot(\text{wt\% Fe}) - 67.10634\cdot(\text{wt\%Nb}) - 28.29854\cdot(\text{wt\% Ni})$$

Phase I

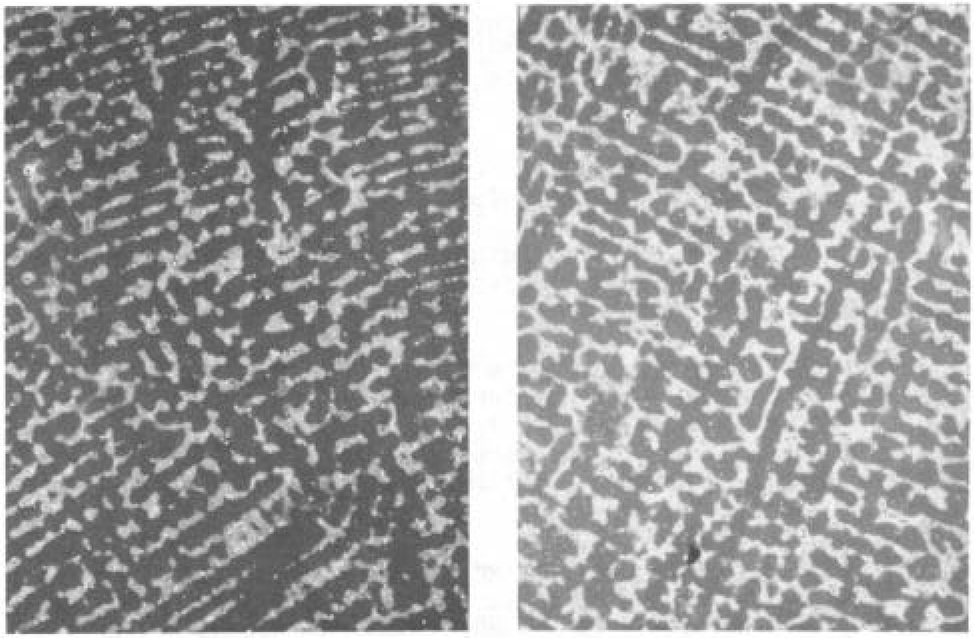
The first phase of this study attempts to correlate the degree of segregation before and after Hot Isostatic Pressing (HIP) samples of the eight modifications of 718.

As-cast Condition of Microstructure. Figure 1 represents the microstructure of the extreme compositions of the eight alloys studied. Both the low Nb, Cr, Fe alloy 47 and the high Nb, Cr, Fe alloy 49 display a composite like structure consisting of bright areas of interdendritic precipitation and dark areas of no precipitation within the dendritic arms. Alloy 47 exhibited the least amount of Nb-rich segregated regions and alloy 49 the most. The remaining six alloys rank between alloy 47 and 49 as shown in Table I. The type and relative amount of precipitated phases per alloy were identified both microstructurally and by x-ray diffraction of extracted residue, presented in Table II. The eutectic Laves, delta plates, globular MC carbides, fine γ'' and γ' precipitates are found in varying degree within the interdendritic regions. Laves is sparse in alloy 47, the low Nb alloy and abundant in alloy 49 the high Nb alloy. The low Fe alloys exhibit the least amount of Laves phase, which suggests that Fe enhances the promotion of Laves and hence segregation within the microstructure.

Table II. Modified As-Cast 718 X-Ray Diffraction Data*

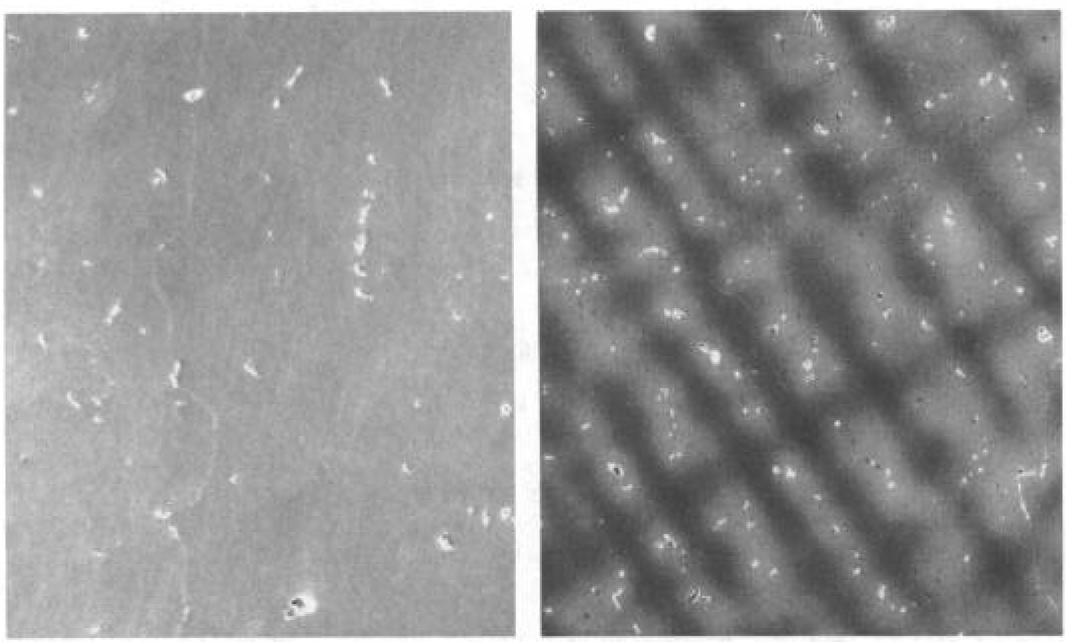
Sample No.	Cr	Fe	Nb	Phases**
47	Lo	Lo	Lo	MC
46	Lo	Hi	Lo	MC-vs, δ w^+
52	Hi	Lo	Lo	MC-vs, δ w^+
53	Hi	Hi	Lo	MC-vs, δ-w^+, L-w^+
48	Lo	Lo	Hi	MC-vs, δ-s
50	Lo	Hi	Hi	MC-s, δ-s, L-m
51	Hi	Lo	Hi	MC-s, δ-s^+, L-m
49	Hi	Hi	Hi	MC-w, δ-vw, L-vs

*Extraction in 10%HCl-M (γ'' does not extract)
**vs = very strong, s = strong, m = medium, w = weak
L = Laves, δ = delta



Alloy 47 (4Nb-14Fe-14Cr) Alloy 49 (5.5Nb-20Fe-18Cr)
Magn. 50X

Figure 1. Microstructure of as cast alloys 47 and 49.



Alloy 47 (4Nb-14Fe-14Cr) Alloy 49 (5.5Nb-20Fe-18Cr)
Magn. 100X

Figure 2. Microstructure of alloys 47 and 49 after thermal HIP + TAG treatment.

Each alloy displayed prominent dense dislocation networks or subgrain boundaries decorated by γ'' precipitation. These networks are more easily observed in the regions of less dense precipitation where the Nb content is lower.

<u>HIP Condition of Microstructure</u>. Except for MC and TiN, HIPing the eight alloys at 2125°F/4 hours completely solutioned all the as-cast precipitation, in both the high and low Nb alloys. After the 1600°F/1 hour TAG thermal treatment the low Nb alloys did not produce visable phase precipitation. However, the high Nb alloys displayed Nb segregation as evidenced by the bright and dark dendritic structures seen in Figure 2.

The standard Nb alloys exhibited a fine γ''/γ' precipitation which appeared more dense in the center of the interdendritic regions, becoming less dense and finally absent in the dendritic arm region. This variation in microstructure demonstrates, by phase precipitation, the incomplete homogenization of Nb throughout the material by the HIP treatment only.

<u>Elemental Analysis</u>. A quantitative elemental scan from the white regions of most dense precipitation to the dark regions of no precipitation reveal a chemical gradient or range which can be used as a measure of segregation.

In each as-cast alloy there is a Nb level at which precipitation begins to take place. This level is consistently 4.15 $\pm$ 0.22%. When the Nb content is above this level, γ'' precipitation occurs, while below this level no precipitation occurs. After HIP + TAG, all the high Nb alloys, exhibit visible precipitation; while, the low Nb alloys show no precipitation. This is not an indication of chemical homogeneity, but rather that when the Nb level falls below 4%, phase precipitation at 1600°F is retarded. A TAG treatment at lower temperatures may be necessary to cause precipitation of γ'' at Nb levels less than 4%.

<u>Phase II</u>

<u>Material Background</u>. The second phase of study was concerned with mechanical properties of the two extreme alloys, 78 and 79. Small heats of alloys 78 and 79 were VIM cast by HTCC into eight oversized test bars. Each bar was heat treated according to General Electric, Co. specifications as follows:

HIP	2125°F/15 KSI for 3-4 hours (GFC)
HOMOGENIZE	2000°F/1 hour
SOLUTION	1750°F/1 hour
AGE	1400°F/5 hours-->1200°F/1 hour

<u>Mechanical Properties</u>. The results of room temperature tensile and stress rupture testing of the standard alloy #79 and modified alloy #78 are presented in Table III. The standard alloy #79 met the General Electric Company cast 718 requirements for tensile and stress rupture.

The modified alloy #78 met the tensile strength, elongation, and reduction requirements of the GE cast 718 specifications, but the yield strength was under the minimum requirements. The first tested stress rupture bar of alloy 78 displayed an exceptionally long stress rupture life. To verify this life, two additional stress rupture tests were run which resulted in very low life.

Table III. Mechanical Properties Data

Room Temperature Tensile Properties

Alloy #	Test #	Additional Thermal Treatment*	0.2 YS MPa	0.2 YS KSI	UTS MPa	UTS KSI	Elong. %	RA %
78	1	0	612	88.7	938	135.9	31.9	38.3
78	2	0	608	88.2	924	133.5	30.4	43.3
78	3	0	1096	159.8	1220	177.1	11.6	23.8
78	4	0	1103	160.6	1255	181.6	19.0	25.2
78	17	1200°F/20 hours	737	106.9	1027	149.1	31.1	32.1
GE Specification (min)			641	93	827	120		

Stress Rupture Properties at 1200°F/90 KSI

Alloy Number	Test Number	Additional Thermal Treatment*	Life (hours)	Elongation %
78	5	0	220.9	5.7
78	6	0	0.3	10.0
78	7	0	866.6**	
79	8	0	642.7	2.5
78	9	0	0.6	10.1
78	10	1650°F/4 hours 1400°F/20 hours	FOL	57.5
78	11	1400°F/20 hours 1200°F/20 hours	5.9	8.4
78	12	1200°F/20 hours	28.7	5.5
78	13	1200°F/20 hours	29.1	7.9
GE Specification (min)			20	

*Heat treatments were imposed on:
2125°F/15 KSI/4 hours
2000°F/1 hour
1750°F/1 hour
1400°F/5 hours + 1200°F/1 hour

**Test was stopped - no failure occurred

Additional #78 samples were tested in stress rupture after supplemental thermal cycles were imposed on the original heat treatment. The bar which was given a 1650°F/4 hour + 1400°F/20 hour exposure exhibited no life. The elongation in this bar was excessive indicating a very ductile material with little or no phase precipitation to provide strength. Additional thermal exposures of 1400°F/20 hours + 1200°F/20 hours and 1200°F/20 hours increased the stress rupture life from 0.3 hours to 29 hours, respectively. The 1200°F/20 hour exposure life of 29 hours meets the GE stress rupture requirements. This result indicates successful precipitation of the strengthening γ'' and/or γ' phases occur at lower temperatures and longer times than for the conventional 718 material.

The results of the stress rupture test suggested that the yield strength of the modified alloy in a tensile test could also be increased by the 1200°F/20 hour heat treatment. Therefore, retest of a bar after the additional 1200°F/20 hour exposure did exhibit an increase in yield strength sufficient to meet the GE tensile specification. It is interesting to note heat treating to increase the yield strength did not decrease the elongation, resulting in a low Nb material which exhibits greater ductility with adequate yield and tensile strength when compared to the conventional 718 alloy. This may be a result of more efficient use of Nb due to a more uniform distribution of the element throughout the microstructure as a result of HIP.

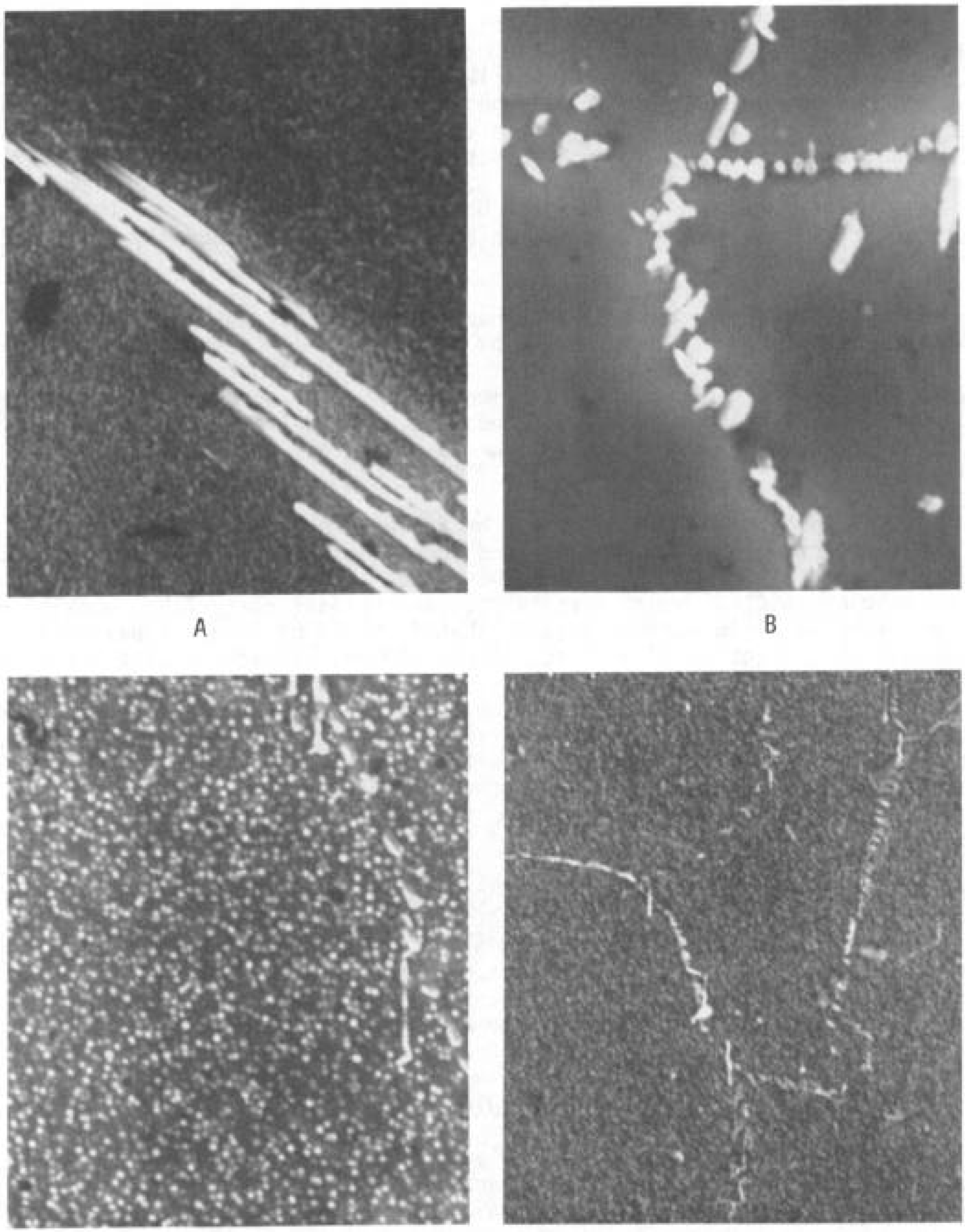
<u>Microstructure</u>. The as-processed conventional alloy 718 exhibited a grain boundary delta phase surrounded by a zone denuded of γ'' and/or γ'. After exposure to 1200°F/90 KSI for 662 hours, Figure 3-A, hours the grain boundary delta phase has elongated and is now surrounded by a very fine γ'' precipitate suggesting additional aging has taken place during test.

The low Nb alloy, in the as-processed condition, yielded 1 stress rupture test to have long life (221 hours) as compared to the two short life (0.3 and 0.6 hour) samples. Comparable microstructures for the low life (0.3 hour) and high life (221 hours) stress rupture bars resulted in no apparent microstructural inconsistencies which would contribute to the observed life variation.

The additional thermal treatment of 1650°F/4 hours + 1400°F/20 hours resulted in the unusual microstructure shown in Figure 3-B. A network of large γ'' plates was found along the subgrain boundaries and in areas of elevated Nb content. There was no precipitation of the fine γ'' and/or γ' phases which explains the 0 hour rupture life of the bar. Apparently 1400°F is too high an age temperature to precipitate the strengthing γ'' in the low Nb alloy.

An additional thermal treatment of 1400°F/20 hours + 1200°F/20 hours, resulted in the microstructure of Figure 3-C. The higher Nb regions dis play a combination of γ'' plates and γ' spheres while the low Nb regions reveal only the γ' spheres. Precipitation of γ'' plates along the subgrain boundaries was evident in both the mixed precipitation region and γ' precipitation region. Comparing the microstructure of this thermal treatment to that of the previous thermal treatment (1650°F/4 hours + 1400°F/20 hours), demonstrates the need for lowering the γ'' and/or γ' phase precipitation temperature when the Nb content of the alloy is 4% or less.

Samples which were given only a 1200°F/20 hour thermal treatment displayed a uniform fine precipitate throughout the microstructure, Figure 3-D. A discrete γ'' disk precipitation was present in the subgrain boundaries. It can be concluded a single 1200°F/20 hour exposure will pre cipitate a very effective γ''/γ' strengthening phase in the low Nb alloy.



A B C D

Figure 3. A: Alloy 79, 5.5Nb-20F3-18Cr, as-processed Magn. 10K
B: Alloy 78, 4Nb-14Fe-14Cr, with additional heat treatment of 1650°F/4 hrs + 1400°F/20 hours Magn. 15K
C: Alloy 78, 4Nb-14Fe-14Cr, with additional heat treatment of 1400°F/20 hours +1200°F/20 hours Magn. 10K
D: Alloy 78, 4Nb-14Fe-14Cr, with additional heat treatment of 1200°F/20 hours Magn. 20K

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Effect of Composition on Segregation Microstructures and Mechanical Properties of Cast Alloy 718
by
S.M. Jones, J.F. Radavich and S. Tian
Page 595

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