ON DEVELOPING A MICROSTRUCTURALLY AND THERMALLY STABLE

IRON-NICKEL BASE SUPERALLOY

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Abstract

Recent advances in the jet engine industry are placing new demands for an alloy which exhibits the excellent fabricability and heat resistant properties of IN718, but which is not hampered by the alloy's relatively low temperature ceiling. This ceiling is imposed by the microstructural instabilities that occur fairly rapidly at these higher temperatures. Seven new iron-nickel base superalloy compositions were designed to study the effect of systematically changing the Al, Ti, and Nb contents on the microstructural stability and long term elevated temperature mechanical properties. Rockwell hardness, tensile, and creep/stress rupture tests were performed on these alloys to determine their extended elevated temperature properties, while optical, scanning electron, and transmission electron microscopy, and x-ray diffractometry were used to analyze their phase stability. Increasing the Al + Nb content and the Al/Ti ratio in the alloy was found to (1) produce more of the stable γ' phase and less of the brittle δ phase, and (2) enhance the mechanical properties of the alloy. Preliminary results indicate that the alloy containing a greater Al + Nb content and Al/Ti ratio is more stable than Inconel 718.

Superalloys 1988
Edited by S. Reichman, D.N. Duhl,
G. Maurer, S. Antolovich and C. Lund
The Metallurgical Society, 1988

Introduction

Inconel 718 (IN718), a precipitation strengthened iron-nickel base superalloy currently containing approximately 5.3 wt. pct. niobium, exhibits adequate strength, ductility and fatigue resistance up to 650°C. Since this temperature corresponds to the medium temperature range in jet engine rotating disk applications, and due to its good fabricability (as a consequence of the higher iron content), IN718 is one of the most used superalloys (1). At present, this alloy accounts for approximately 35 pct. of all wrought superalloy production. Recent advances in the jet engine industry are placing new requirements on turbine engines. These include a need for equal or greater strength at temperatures above the heat resistance of the otherwise highly fabricable IN718-type alloy.

Two precipitating phases are responsible for the high temperature mechanical properties of the matrix gamma (γ) phase. These two phases are gamma prime (γ') and gamma-double prime (γ'') (2,3). Gamma prime, the first of the two phases to precipitate during heat treatment, is a coherent, ordered $Ni_3(Al,Ti,Nb)$ face centered cubic structure. Gamma-double prime, which is reported to nucleate and coarsen on the γ' particles and in the matrix, is a coherent, but misfitting and ordered metastable $Ni_3(Nb,Al,Ti)$ body centered tetragonal structure (4).

Not welcome in this alloy is the brittle, needle-like delta (δ) phase. This phase is an incoherent, orthorhombic nickel and niobium rich phase that precipitates as a needle-like structure after extended times at elevated temperatures. Because of the crystal symmetry between the close packed (112) plane of the γ'' phase and the close packed (010) plane of the δ phase, it is believed that the γ'' particles act as nucleation sites for the δ phase (5). It is this δ transformation, accompanied by the overaging and partial dissolution of the precipitation strengthening phases that sets the temperature-time limits for the engine application of IN718 (6,7).

The goal of this study is to reduce the amount of overaging of the γ' and γ'' phases, and to increase the transformation time to form δ. The strategy involves systematically varying the chemistry of those elements responsible for forming these precipitating phases; i.e., aluminum, titanium, and niobium. The most promising modification appears to point at modifying the alloy in the direction of a higher (Al+Ti)/Nb atomic ratio. At present in Inconel 718 this ratio stands at approximately 0.7. Preliminary experimental results (8) suggest that allowing the atomic percent of aluminum and titanium to equal that of niobium may result in a more thermally stable γ'', due to the greater fraction of γ'' particles coarsening on γ'. Increasing the amount of aluminum and titanium should result in more γ' and especially in more γ' surface area for the γ'' to nucleate and grow. By increasing the number of γ'' nucleation sites, more γ'' particles should form. In doing so, this would produce finer sized γ'' particles upon reaching equilibrium, which could result in a reduction in the driving force to form δ.

Three sets of alloys were produced. The first alloy series examines the effect of increasing the (Al+Ti)/Nb ratio by increasing the Al+Ti content, while maintaining the niobium level at 5.25 wt. pct. The second alloy series compares the effects of aluminum and titanium on the stability of each phase by increasing the Al/Ti ratio from 0.86 to 1.67 while maintaining the (Al+Ti)/Nb ratio and titanium content. The third set, which was produced after an intensive investigation of the first two sets, follows the strategy of the second series by increasing the Al/Ti ratio to 1.78; however, this series also studies the effect of reducing the Ti content in the alloy. Work on this new series of alloys is still in progress, so not all comparative results have been acquired at this point.

Procedure

A master billet of SUPER IN718 was supplied courtesy of The Wyman-Gordon Company to be used as the base material to produce the standard and seven modified alloys used in this study. This billet was cut into equally sized pieces. Each piece was vacuum induction melted (VIM) courtesy of the Special Metals Corporation into 15 lb ingots and alloyed to the desired chemistry, see Table I. The alloy designated as IN718-1 is the standard "SUPER" IN718 alloy.

Table I. Alloy Chemistries (wt. %)

Alloy	Al	Ti	Nb	Ni	Fe	Mo	Cr	C	(Al+Ti)/Nb	Al/Ti
IN718-1	0.46	0.95	5.26	bal	18.25	3.05	18.00	0.031	0.65	0.86
4	0.63	1.34	5.28	bal	18.30	3.05	17.80	0.028	0.90	0.83
9	0.53	0.96	4.32	bal	18.30	3.02	18.15	0.31	0.85	0.98
10	0.68	0.97	4.91	bal	18.30	3.03	18.10	0.031	0.86	1.24
11	0.87	0.96	5.42	bal	18.00	3.02	17.80	0.031	0.90	1.61
11b	0.85	0.95	5.47	bal	17.97	3.05	17.70	0.035	0.87	1.59
12	0.94	0.96	5.72	bal	17.83	3.05	17.53	0.032	0.89	1.74
13	0.87	0.90	5.38	bal	18.05	3.04	17.80	0.035	0.88	1.72
14	0.86	0.86	5.66	bal	17.95	3.06	17.70	0.035	0.82	1.78

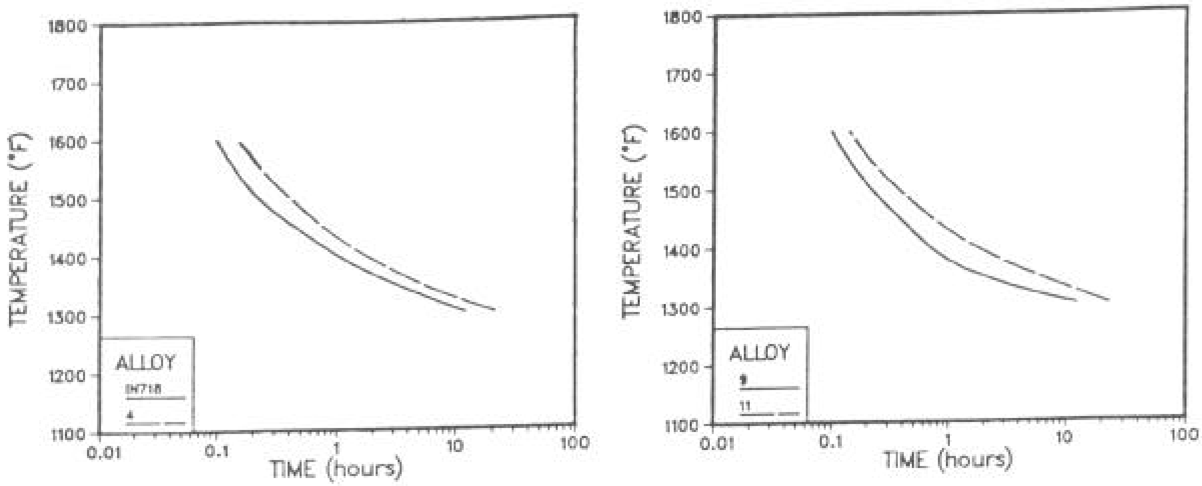
* Also contains approximately: 0.02 Mn, 0.11 Si, 0.02 Ta, 0.003 B, 0.002 S, 0.006 P, 0.006 Cu, 8 ppm Mg, 20 ppm Sn

A homogenization practice first had to be determined for the new experimental alloys. This was done by a solutionization and hot working step. The forging temperature window was determined for each alloy by heat treating cut pieces and optically analyzing them for any incipient melting (IMP) or δ phase (9).

Forty three pieces measuring 1.5 cm^2 x 0.15 cm were cut from each billet. Following this, they were solutionized at 1110°C for six hours, heat treated for various times and temperatures, and quenched. The precipitated phases were then extracted from each sample in a 1 pct. citric acid, 1 pct. ammonium sulfate, distilled water solution, and the extracted residue was filtered from the solution. X-ray diffraction patterns were produced from each extracted residue and the phases identified. In addition, diffraction patterns were produced for these alloys in their fully-aged condition to (1) determine whether any extraneous phases had formed due to these modifications, and (2) measure the changes in the relative amount and lattice parameters of each phase.

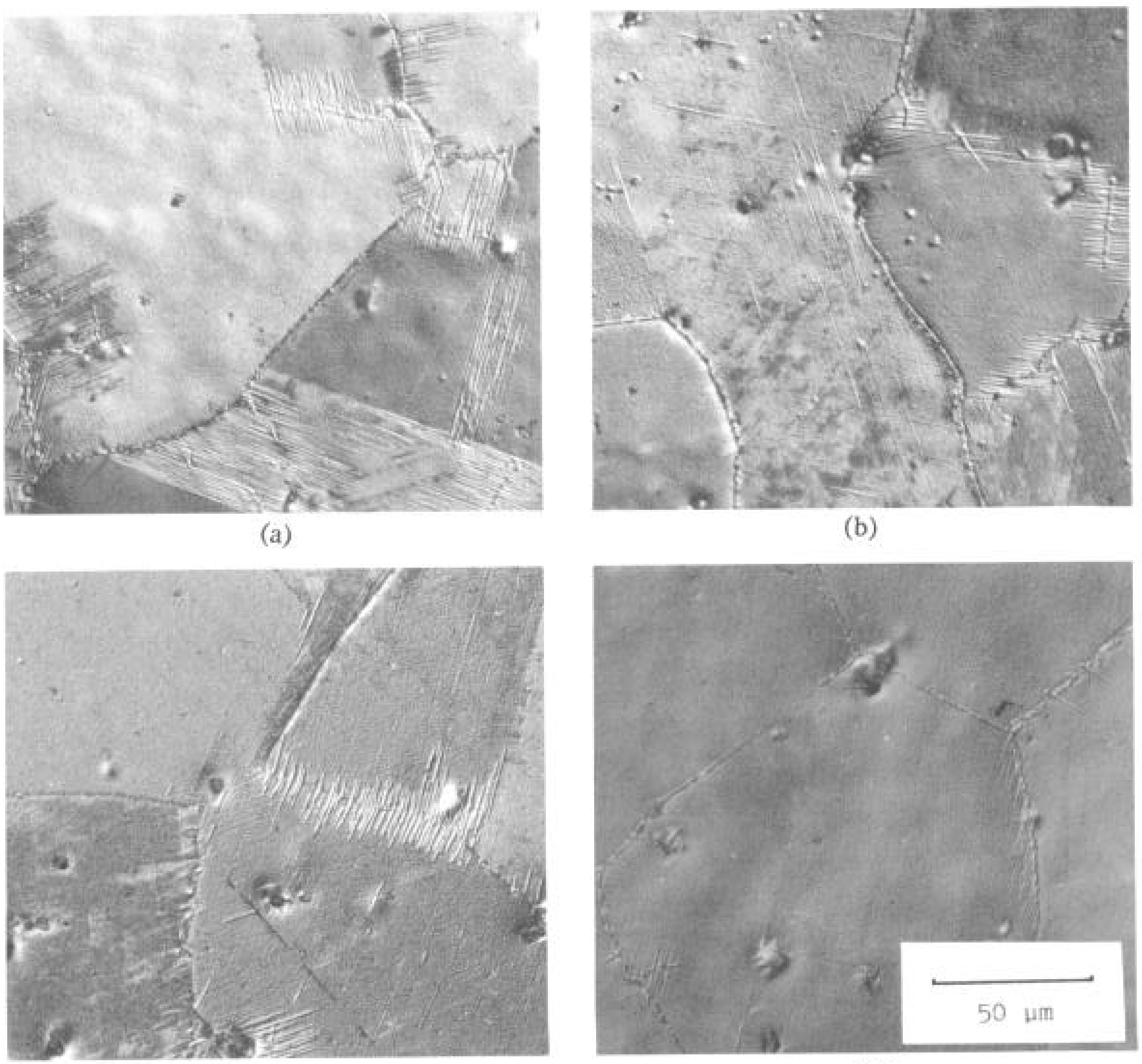
Microstructural information for this study was determined using optical, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The samples were electropolished in a 5 pct HCl, 5 pct $HClO_4$, ethanol solution, and etched with Kalling's waterless reagent. The TEM specimens were jet polished at -10°C in a 10 pct $HClO_4$, 30 pct n-butanol, ethanol solution.

Following commercial specification tests, ambient temperature, and 650°C



(a) (b)

Figure 1 - Temperature-Time-Transformation Curves for Alloys (a) 1 and (4), and (b) 9 and 11



(a) (b)

(c) (d)

Figure 2 - Optical Micrographs of alloys (a) 1, (b) 4, (c) 9, and (d) 12 Heat Treated for 600 Hours at 760°C

tensile tests were performed on these alloys by the Wyman-Gordon Company.

Two types of tests were employed to examine the mechanical properties of these alloys after long exposure times at elevated temperature. The first of these, the Rockwell hardness test, is a simple and effective test used to indicate any changes in strength upon varying heat treatments which could be related to a change in microstructure. In this case the alloys were solutionized at 1110°C for six hours and heat treated at 760°C for times ranging from 1 to 600 hours. The second type of test consisted of comparing the results from two sets of 705°C creep-stress rupture tests. The first set was tested after the standard heat treatment practice, while the second set was tested after being heat treated at 732°C for 1000 hours following the standard heat treatment. Both sets were machined into creep bars after receiving their specific heat treatments.

Results

The diffraction patterns produced for the alloys in their fully-aged condition reveal that the alloys with the highest (Al+Ti)/Nb and Al/Ti ratios contain less δ and more γ' (9). Increasing the Al/Ti and (Al+Ti)/Nb ratios in the alloy causes an increase in the lattice parameters of the γ' and γ" phases, most notably in the c-axis of the γ" phase, see Table II. In addition, increasing these ratios delays the formational times of the δ phase, as seen in the δ Temperature-Time-Transformation curves presented in Figure 1.

Table II. X-Ray Lattice Parameters of γ' and γ' Phases

Alloy	γ' a-axis (Å)	γ'' a-axis (Å)	γ'' c-axis (Å)
1	3.606	3.626	7.416
4	3.607	3.625	7.416
9	3.606	3.625	7.410
10	3.607	3.626	7.417
11	3.607	3.626	7.423
11b	3.607	3.626	7.422
12	3.608	3.627	7.429
13	3.606	3.625	7.422
14	3.607	3.629	7.418

Optical micrographs of selected alloys heat treated at 760°C for 600 hours are presented in Figure 2. It can be seen that the alloys with the highest (Al+Ti)/Nb and Al/Ti ratios, i.e., alloys 4 and 12, contain the least amount of δ.

Figure 3 reveals dark-field TEM micrographs of alloys 1, 4, 9, and 12 following a 100 hour heat treatment at 760°C. The average particle sizes of the γ' and γ" phases are given in Table III. In addition, the volume fractions of the γ' and γ" in these alloys were determined using the thin foil method, Table IV (10).

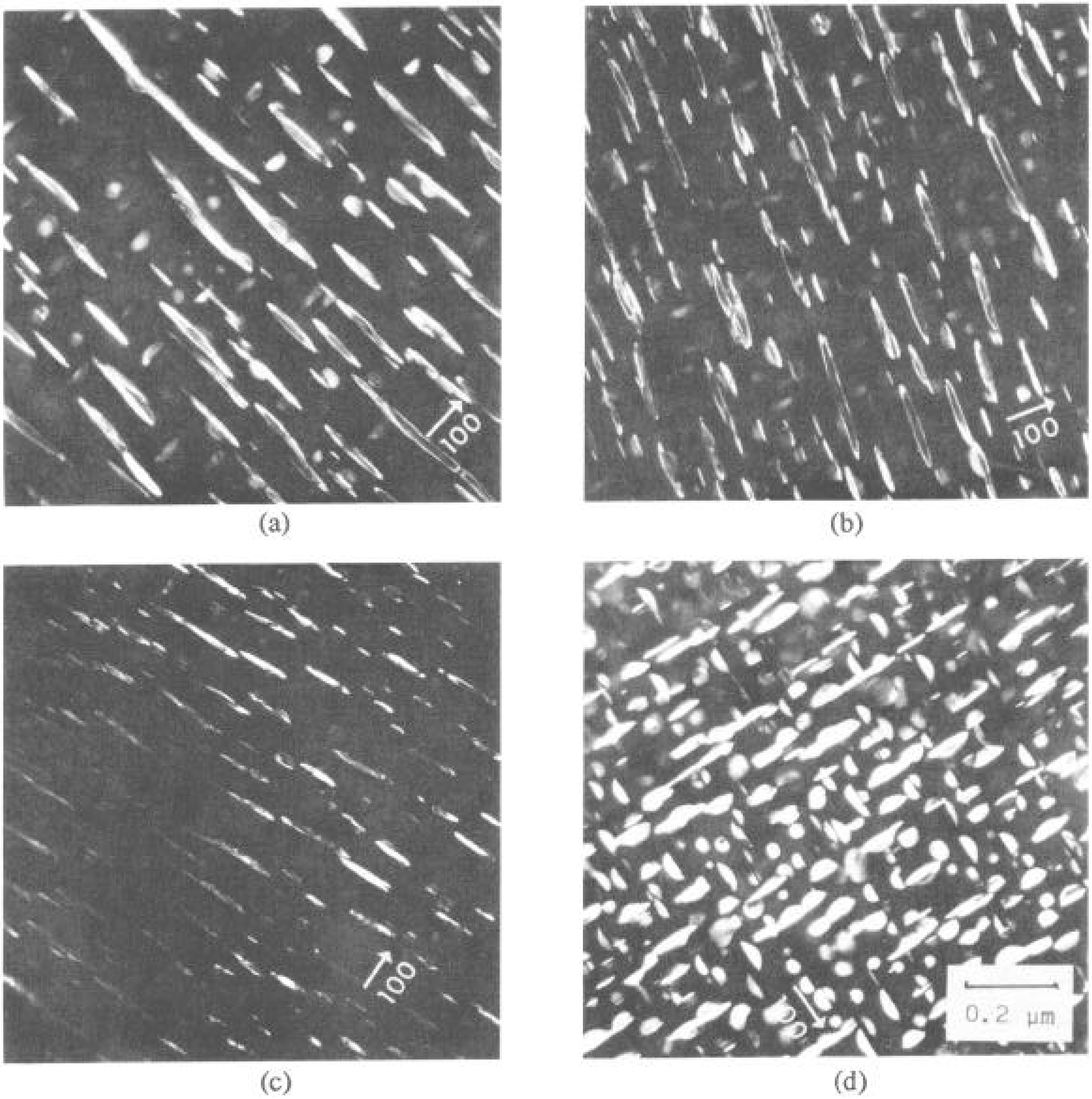
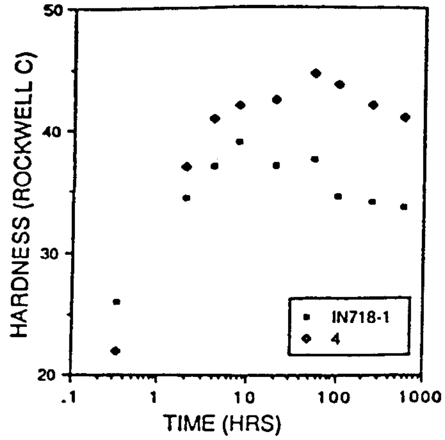


Figure 3 - Dark-field images of alloys (a) 1, (b) 4, (c) 9, and (d) 12 heat treated at 760°C for 100 hours. Dark-field micrographs were taken with the (100) ($\gamma' + \gamma''$) orientation.

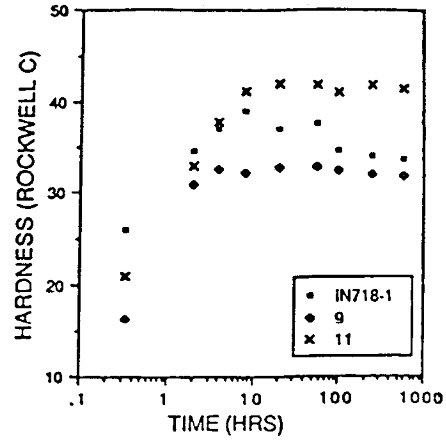
Increasing the Al/Ti and/or the (Al+Ti)/Nb ratios in the alloy increases the size and volume fraction of the γ' phase, and decreases these values for the γ'' phase. The number of γ'' particles nucleating on γ' particles was found to increase by an order of magnitude with these increased ratios. In addition, Table III reveals that increasing the Al/Ti ratio causes a decrease in the γ'' particle length/thickness ratio.

Ambient temperature, and 650°C tensile tests are given in Table V. Figure 4 reveals the results from the Rockwell hardness tests, and Table VI gives the 705°C creep-stress rupture results from the alloys tested in their initial commercially heat treated condition, and also following a 1000 hour exposure at 732°C.

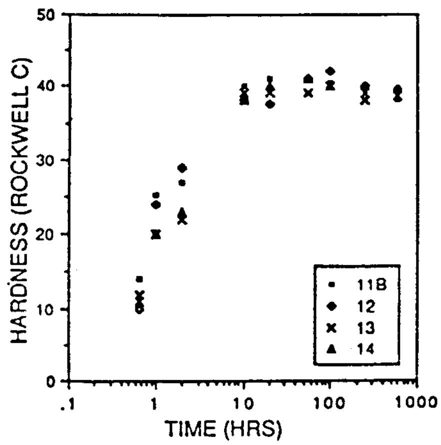
Increasing the Al/Ti and (Al+Ti)/Nb ratios enhanced the yield and ultimate tensile strengths of the alloy at both temperature ranges studied. The results from the Rockwell hardness and 732°C creep/stress rupture tests indicate a marked improvement in long time elevated temperature mechanical properties when the Al/Ti and/or the (Al+Ti)/Nb ratios are increased in the alloy.



(a)



(b)



(c)

Figure 4 - Hardness Values of Alloys Heat Treated at 760°C

Table VI. 705°C Creep/Stress Rupture Results

(70 KSI)

	Initial Heat Treatment		+ 732°C - 1000 Hour Exposure	
Alloy	Rupture Life (hours)	ε (%)	Rupture Life (hours)	ε (%)
1	210.9	13.3	3.0	21.8
4	187.6	16.7	20.6	21.6
11	185.8	17.2	18.2	36.6

Discussion

One of the objectives in this study pertains to reducing the amount of overaging of the γ'' particles in order to stabilize the mechanical properties of the alloy. By increasing the Al/Ti and/or the (Al+Ti)/Nb ratios in the alloy, the average length of the γ'' particles was reduced by approximately 50 percent. The reason for this reduction in γ'' size is twofold. First, the expansion of the γ'' lattice parameters, especially the c-axis, increases the misfit of this phase within the γ matrix. As a result, it becomes more difficult for the γ'' to nucleate within the γ at a given temperature. This difficulty is apparent in the observation of a greater fraction of γ'' particles seen nucleating on γ' particles. The γ' particles have a larger lattice parameter than the matrix, and, therefore act as heterogeneous nucleation sites for the γ'' particles. Second, the greater Al content increases the amount of γ' in the alloy. This γ' may be richer in Nb, which would deplete the amount of Nb available in the matrix to form γ''.

The second objective of this study was to reduce the driving force to form δ at elevated temperatures. Figure 1 shows that increasing the Al/Ti and/or the (Al+Ti)/Nb ratios in the alloy increases the reactional times to form δ. As previously mentioned, it is believed that the close packed (112) planes of the γ'' can act as the (010) habit plane for the δ phase. Increasing the Al/Ti and (Al+Ti)/Nb ratios not only decreases the volume fraction of γ'', but also reduces the average size and thickness of these particles. As a result there is a decrease in γ'' surface area for the δ to nucleate. Another explanation for the reduction in the amount of δ could be the result of the increased volume fraction of γ' in the alloy.

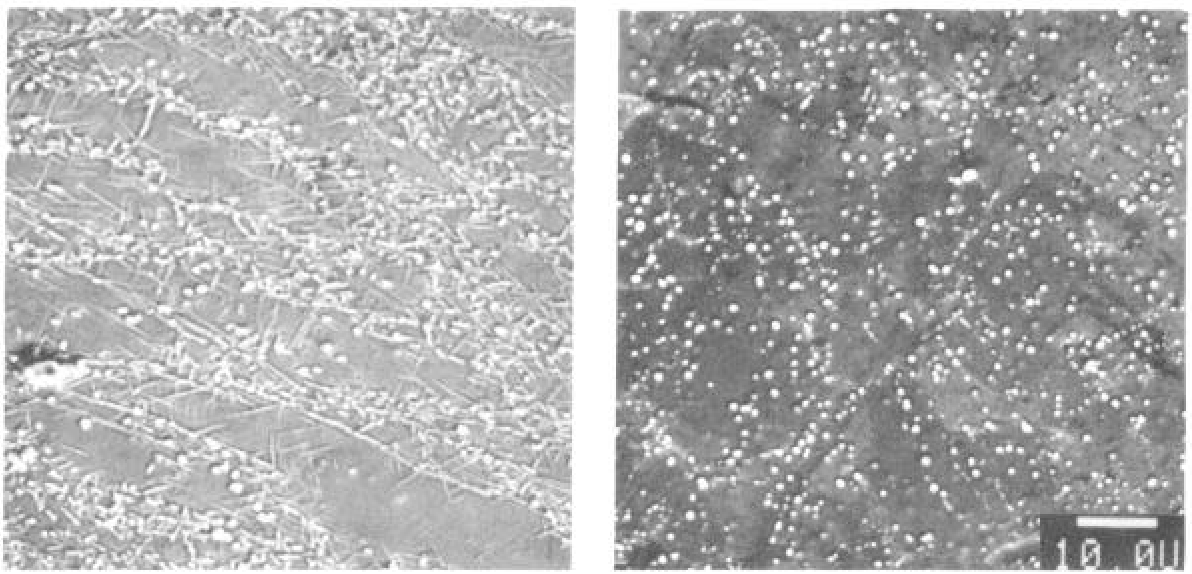


Figure 5 - Scanning Electron Micrographs of Alloys (a) 1, and (b) 11 after 1000 Hour exposure at 732°C

The mechanical results reveal that the properties of the alloys following the commercial heat treatment are somewhat enhanced with the increased Al/Ti and/or (Al+TI)/Nb ratios. These results are not surprising, since the loss in γ'' volume fraction is accompanied by an increase in γ' fraction.

The results from the Rockwell hardness and the 705°C creep/stress rupture tests show a large improvement in the long term elevated temperature mechanical properties of the alloy when the Al/Ti and/or the (Al+Ti)/Nb ratios were increased. The microstructure of the standard IN718 and alloy 11 following a 1000 hour heat treatment at 732°C is presented in Figure 5. It can be seen that the standard alloy is filled with δ, while alloy 11 contains only a trace amount of this brittle phase. Considering the effect on the size of the γ'' particles and the amount of δ in the alloy due to these modifications, these results are not surprising.

Concluding Remarks

These preliminary results indicate that increasing the Al/Ti ratio and/or the

Al+Ti content above that of the original IN718 results in a more thermally and mechanically stable alloy. This could be due to the higher volume fraction of γ', the smaller and more misfitting γ'' particles, and/or the reduced amount of δ in the alloy. These results also indicate that more of the high temperature heat resistant niobium can be added into the alloy without any corresponding loss in stability.

The second phase of this alloy design program is currently under way, which will study the properties of a larger, commercial-sized ingot of the most stable alloy to date, alloy 12. The goal of this phase is to produce a more thermally stable to replace the existing "SUPER" IN718 alloy.

Acknowledgements

This study was made possible by a gift grant from The Niobium Products Company, a subsidiary of Companhie Brasileira de Metallurgia e Mineracao (CBMM). We thank Harry Stuart and E. A. Loria for their interest and monitorship. We thank The Wyman-Gordon Company for the material and mechanical working of the alloys, in particular, Steve Reichman and W. H. Couts for their technical advice. We thank The Special Metals Corporation for their assistance in casting the original alloys.

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