

STRUCTURE/PROPERTY EFFECTS OF SUBSTITUTIONS

FOR REDUCED COLUMBIUM IN ALLOY 718

K.R. Ziegler* and J.F. Wallace+

*Argo-Tech Corporation
Cleveland, Ohio

+Department of Materials Science and Engineering
Case Western Reserve University
Cleveland, Ohio

ABSTRACT

Columbium, chromium, cobalt and tantalum have been identified as metals vital to the aerospace industry. This investigation was undertaken to determine the effects of substitutions of other elements to maintain the properties of Alloy 718. The room and elevated temperature tensile properties, and the stress-rupture properties were examined for a series of 13 alloys with substitutions of molybdenum, tungsten, vanadium, titanium and boron for the reduced columbium.

The substitution of molybdenum and tungsten and vanadium were unable to recover the properties of the original alloy. The structure and properties indicated that these additions did not alter the volume fraction of γ'' . Based on literature information, titanium and columbium additions were selected to substitute γ' precipitation for the decreased γ'' precipitation while maintaining the slow coarsening rate needed in Alloy 718. The result was an alloy with 3.89Cb + 1.27Ti, strengthened by both γ'' and γ' . The hardening rates of the 3.89Cb + 1.27Ti alloy was comparable to an alloy with 3.2Cb, indicating a similar precipitation sequence. Microstructural analysis as a function of the aging temperature indicated that the γ' phase precipitated first while γ'' precipitation followed but provided the greater contribution to the room temperature hardness and strength.

The strength of the standard alloy decreased rapidly at temperature above 540°C, and the benefit of boron diminished at the higher temperatures. The higher titanium contents prevented a dramatic loss in strength at temperatures up to 650°C. The stress-rupture tests indicated that similar stress-rupture strength can be expected for the Alloy 718 and the alloy with 3.89Cb + 1.27Ti.

Introduction

Columbium, chromium, cobalt and tantalum have been identified as metals that are vital to the aerospace industry. Over 90% of each of these metals is imported from sources that are viewed as unreliable. Historically this has resulted in large price and supply fluctuations that are potentially harmful to the U.S. national defense. The National Materials and Minerals Policy was adopted to focus attention on the price and supply fluctuations of these strategic metals. This policy also established a program to explore the role of these materials in aerospace materials and identify alternative materials and processing techniques to reduce the dependence on these metals (Ref.1).

Nickel and nickel-iron based superalloys containing γ' precipitation, which are spherical to cuboidal in shape, rely on strengthening mechanisms that are a function of composition, heat treatment and temperature (2,3). These alloys use significant aluminum and titanium additions to generate γ' precipitation. In nickel-iron based superalloys that are γ'' strengthened, such as Alloy 718, coherency strains between the matrix and the precipitate are the primary strengthening mechanism (4-6). The large strain fields surrounding each precipitate inhibit nucleation of more γ'' , limiting the γ'' phase in Alloy 718 to about 20 volume percent, while typical superalloys can have as much as 60 volume percent γ' . The coherency strains surrounding the disk shaped γ'' precipitates diminish at higher temperatures, limiting the service temperatures of Alloy 718. The γ'' phase in Alloy 718 is a BCT precipitate that results from substantial additions of columbium (typically 5.0 to 5.5 weight percent) to a nickel-iron base composition (7). The alloy also contains about 1.2 weight percent aluminum and titanium, resulting in some γ' precipitation. There are some indications that γ' nucleates γ'' precipitation (8).

This investigation was undertaken to determine the effects of removing large amounts of columbium from Alloy 718 and to investigate the substitution of other elements to maintain the properties of the standard alloy. The design of an alloy that will perform similar to Alloy 718 with reduced columbium contents can proceed along several paths: solid solution strengthening alloying can replace the columbium; the reduced columbium can be replaced with other precipitate-forming elements; the remaining columbium can be made available for γ'' formation through composition changes. Each of these strategies was evaluated in this program.

Procedure

A series of thirteen alloys, with columbium levels below the standard Alloy 718, were investigated as substitutes for the standard alloy (Table I). Alloy 1a is the standard Alloy 718 composition. Alloy 1b is the same with additions of boron. Alloys 2a through 2f and alloys 3a through 3d contain substitutions of molybdenum, tungsten, vanadium and boron for the reduced columbium.

There is some evidence that W, V, and Mo partition to the strengthening precipitates in superalloys, and W and Mo provide potent solid solution strengthening. In addition, V forms a stable MC carbide while W and Mo contribute to the formation of $M_{23}C_6$ and M_6C grain boundary carbides (9). The boron was selected to improve room and elevated temperature strength and ductility by eliminating continuous grain boundary carbide films (10).

Table I. Alloy Composition

Alloy	Cb	Ti	<u>Weight Percent</u>		V	B
			Mo	W		
1a	5.31	0.87	3.10			
1b	5.30	0.87	3.10			0.04
2a	3.20	0.87	3.10			
2b	3.20	0.87	3.10			0.04
2c	3.10	0.87	5.80			
2d	3.10	0.87	5.80			0.04
2e	3.00	0.87	2.99	3.00		
2f	3.00	0.87	3.00	3.00	0.9	
3a	1.10	0.87	3.10			
3b	1.10	0.87	5.80			
3c	1.10	0.87	3.00	3.00		
3d	1.10	0.87	3.00	3.00	0.9	
4	3.49	1.09	3.00			
5	3.89	1.27	3.00			

Table II. Solution and Age Heat Treatments

<u>Solution Heat Treatment</u>		<u>Age Heat Treatment</u>	
Temperature (°C)	Time (Hrs)	Temperature (°C)	Time (Hrs)
930	2	650	100
980	2	705	50, 100
1040	2	760	25, 50, 100
1095	2	815	10, 25
		870	5, 10

Table III. Room Temperature Tensile Properties

Alloy	Cb (wt.%)	0.2% Offset Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)
1a	5.31	842	1287	16.5
1b	5.30	936	1369	19.4
2a	3.20	532	1091	30.8
2b	3.20	671	1157	24.7
2c	3.10	531	1100	36.5
2d	3.10	616	1138	28.8
2e	3.00	631	1156	30.0
2f	3.00	665	1162	27.7
3a	1.10	448	956	21.0
3b	1.10	521	963	20.5
3c	1.10	486	978	23.8
3d	1.10	517	969	21.9
4	3.49+Ti	752	1180	25.0
5	3.89+Ti	869	1240	23.5

After a review of previous work, several solution and age heat treatment temperatures were selected for study. The standard Alloy 718 was given the typical heat treatment cycle while those for the alternative alloys were selected to highlight the microstructures of each alloy. As such the hold times at the various temperatures were extended (Table II). The solution treatments were varied to demonstrate the effect of temperature on alloy homogenization, grain size, and solvus points, while higher than feasible age temperatures were included to examine the effects of over-aging on the alloy structure. All heat treatments were conducted in air with 0.5 inch cube samples cut from hot rolled plates of each alloy.

Hardness tests (Rc), which have been shown to correlate with the yield strength, were used to screen the alloys for the heat treatment cycle that developed the proper microstructure. Tensile tests were carried out at room temperature and at 540, 595 and 650°C. Stress rupture tests, using stresses to produce failure in Alloy 718 in 50 and 100 hours, were conducted at 595 and 650°C. The microstructural analysis of the alloys was carried out with a scanning electron microscope and the strengthening and minor phases were identified by x-ray diffraction of extracted phases.

Results

Analysis of the hardness data and the microstructures of the standard and alternative alloys indicated that the following heat treatment cycle provided the best indication of the range of phases possible in these alloys:

Alloys 1, 2a-f, 3a-d

Solution heat treated 1040°C; 2 hours; air cool
Age heat treated 705°C; 100 hours; air cool

Alloys 4, 5

Solution heat treated 980°C; 2 hours; air cool
Age heat treated 705°C; 100 hours; air cool

The solution temperatures for the lower columbium alloys 2, 3, 4 and 5 were similar to that for Alloy 718. The additions of molybdenum, tungsten, vanadium and boron had no discernable effect on the solution of the γ'' , γ' , δ or the TCP phases. The increased titanium content of alloys 4 and 5 resulted in higher volumes of titanium-rich MC carbides. MC carbides and minor amounts of Laves phase and were present in all the alloys. The M_3B_2 boride was detected in alloy 1b (Alloy 718 + B).

The hardness and tensile properties of alloys 2 and 3 clearly demonstrated the strong dependence of Alloy 718 on the columbium contents and γ'' precipitation. Figure 1 shows the linear relationship between the strength of alloys 1a, 2a and 3a and the columbium content. The complete room tensile test results are shown in Table III. The substitutions of molybdenum, tungsten, vanadium had a minor effect on the mechanical properties. Additions of boron improved both the strength and ductility of the standard alloy and lower columbium-containing alloys. Figure 1 also shows that substitutions of titanium for the reduced columbium, alloys 4 and 5, enhances the mechanical properties of the alloy.

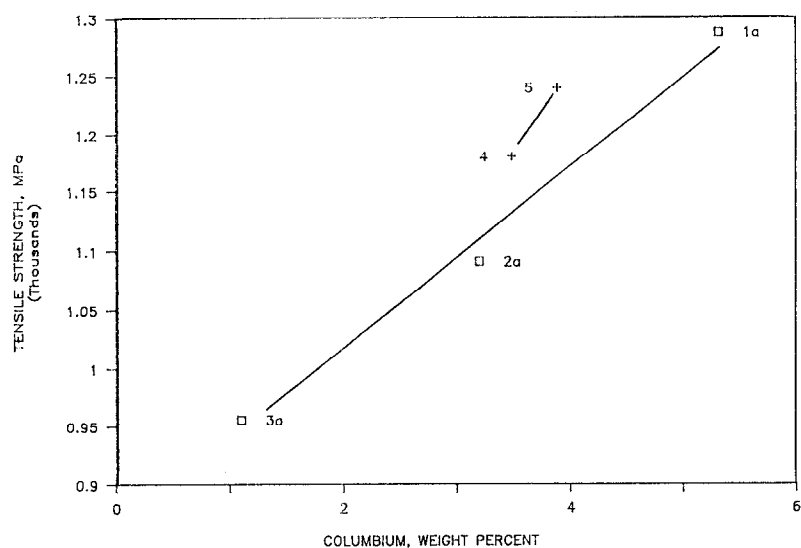


Figure 1 - Tensile strength as a function of columbium.

Table IV. Elevated Temperature Tensile Properties

Alloy	Test Temperature(°C)	0.2% Offset Y.S. (MPa)	Ultimate T.S.(MPa)	% Elongation
1a	540	991	1207	7.7
1b		1087	1251	9.8
4		733	990	24.0
5		833	1059	20.0
1a	595	949	1190	10.7
1b		996	1198	12.4
4		728	977	21.5
5		825	1045	19.5
1a	650	837	1053	12.8
1b		874	1092	16.5
2a		578	816	20.0
2b		635	912	23.5
3a		444	696	23.8
3b		454	761	18.9
4		718	945	16.0
5		810	1022	14.0

Table V. Stress Rupture Properties

Alloy	Test Temperature(°C)	<u>50 hour test</u>		<u>100 hour test</u>	
		Stress(MPa)	Life(Hrs)	Stress(MPa)	Life(Hrs)
1a	595	827	20.1	797	88.7
1b			164.8		400**
4			60.1		87.5
5			147.9		430.8
1a	650	655	25*	620.5	100.0
1b			100*		200.5
4			33.2		75.0
5			116.4		175.5

* Estimated

** Test terminated

Table VI. Columbium Available for γ'' Precipitation

Alloy	Atomic % Cb	Atomic % Cb forming CbC	% Decrease in Cb Available for γ'' Precipitation
1a	3.33	0.20	4.8
2a	2.06	0.19	7.3
3a	0.72	0.15	16.7
4	3.18	0.19	4.7
5	3.43	0.19	4.4

The tensile properties of the alloys were determined over a range of elevated temperatures to indicate the effects of the various microstructural components on strength and ductility (Table IV). The strength of the standard alloys decreased rapidly at temperatures above 540°C, and the benefit of boron diminished at the higher temperatures. The higher titanium contents in alloys 4 and 5 prevented a dramatic loss in strength at temperatures up to 650°C.

The stress-rupture tests concentrated on comparing the alloys with titanium substitutions with the standard alloy. Table V indicates that similar stress-rupture strength can be expected with Alloy 718 and the alloy with the highest titanium levels (alloy 5).

Discussion

As described in the Introduction, the design of an alloy that will perform similar to Alloy 718 with reduced columbium contents can proceed along several paths: substitution of solid solution alloying can replace the columbium; the reduced columbium can be replaced with other precipitate-forming elements; the remaining columbium can be made available for γ'' formation through composition changes. Each of these strategies will be discussed below.

The substitution of solid solution strengthening elements, such as the molybdenum and tungsten additions in alloys 2c-f and 3c-d, could expand or contract the matrix lattice, altering matrix- γ'' mismatch (11). The resulting change in coherency strains alters the precipitation characteristics and strengthening mechanism of Alloy 718. These changes could be controlled to reduce the coherency strains, making the alloy more stable at higher temperatures, but sacrificing the slow γ'' coarsening rate. At longer aging times the hardness of standard and modified alloys in this study were the same, indicating the molybdenum and tungsten additions did not significantly alter the hardening rate. This also indicated the relative ineffectiveness of solid solution strengthening elements as substitutes for columbium in Alloy 718.

A recently documented technique to enhance γ'' precipitation in Alloy 718 suggested that since columbium is such a potent carbide former, the columbium available for γ'' formation is reduced by the stoichiometric requirements of MC carbides (12). Table VI indicates that the amount of columbium remaining in the alloys after the formation of MC carbides. This table does not account for the columbium used to form carbides with titanium and vanadium. The addition of vanadium to displace the columbium in the carbides was not effective. The structure and properties of alloys 2f and 3d indicate that vanadium additions did not alter the volume fraction of γ'' . The reduced MC carbide levels may also affect the thermomechanical processing of the alloy (13).

The substitution of elements such as titanium could maintain the original strength of Alloy 718 by supplementing the γ'' precipitation with γ' precipitation. The proper balance of titanium and columbium must be maintained to avoid changing the precipitation characteristics of the alloy. Literature information indicated a strong effect of the (Ti+Al)/Cb ratio on the precipitate structure. Figure 2 is a plot of the effect of (Ti+Al) on the dominant strengthening precipitates in alloys from this study and from the literature (14-18). Figure 3 indicates the effect of titanium and columbium on the overaged phase. The overaged phase in alloys 1 and 2, determined by x-ray diffraction, was the δ phase.

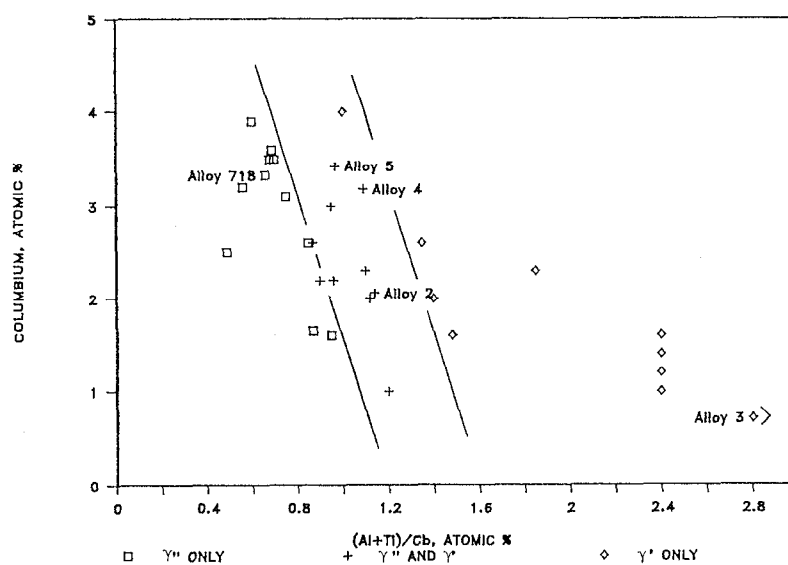


Figure 2 - Effect of Cb, Ti and Al on the strengthening precipitate.

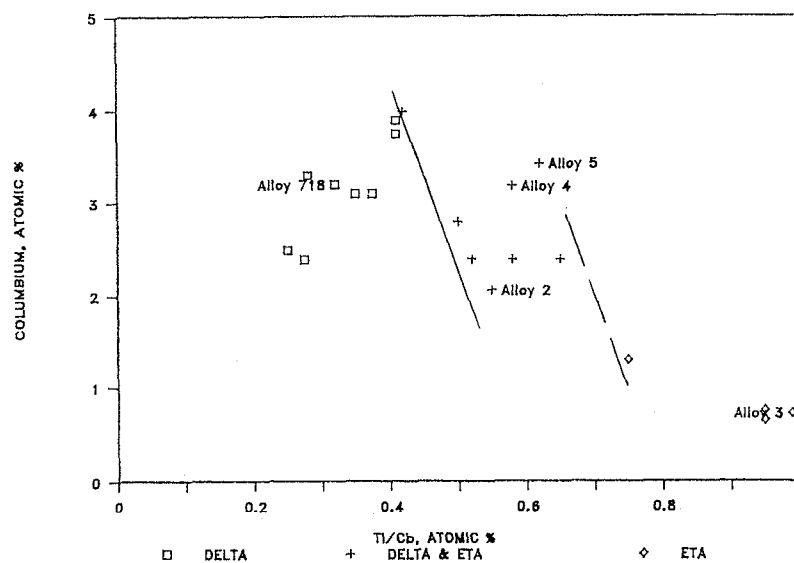


Figure 3 - Effect of Cb and Ti on the overaged phase.

Table VII. Effect of Al, Ti and Cb on Strengthening Phases

Alloy	Cb	Weight %		Atomic %		Precipitates Detected (Strength; Overage)
		Al	Ti	Ti+Al Cb	Ti Cb	
1a	5.31	0.55	0.87	0.66	0.32	γ'' ; δ
2a	3.00	0.55	0.87	1.14	0.55	$\gamma'' + \gamma'$; δ
3a	1.10	0.55	0.87	3.36	1.69	γ' ; ?
4	3.49	0.53	1.09	1.09	0.58	$\gamma'' + \gamma'$; ?
5	3.89	0.52	1.27	0.97	0.62	$\gamma'' + \gamma'$; ?

Titanium forms the Ni_3Ti η overaged phase. While the effect of the overaged phase on the properties is not known, the δ or $\delta + \eta$ phases were retained in the design of the alternative alloys.

Table VII lists the primary elements responsible for precipitation and their effects on the metastable strengthening and the stable phases of alloys 1, 2 and 3. Figure 2 indicates that alloy 1 should have predominantly γ'' , alloy 2 should be a combination of $\gamma'' + \gamma'$ and alloy 3 should be predominantly γ' . The x-ray analysis of the extracted phases and Figures 4, 5 and 6 confirmed the precipitation characteristics of these alloys. With the ratios in Table VII, and using Figures 2 and 3, titanium and columbium levels were selected for alloys 4 and 5 to substitute precipitation for the decreased γ'' precipitation while still maintaining the slow coarsening rate needed in Alloy 718. The result was an alloy strengthened by both γ'' and γ' . Microstructural and x-ray analysis of alloys 4 and 5 confirmed the effect of the $(\text{Ti} + \text{Al})/\text{Cb}$ ratio on the strengthening phase. Figures 7 and 8 confirmed that the precipitates in alloy 5 were a combination of disk and spherical particles (γ'' and γ'). The volume fraction of $\gamma'' + \gamma'$ was less than expected, perhaps because of the relatively high carbon content of alloys 4 and 5.

The hardening rate of the reduced columbium alloys were monitored to insure the precipitation characteristics of Alloy 718 were maintained in the alternative alloys (Figure 9). The hardening rates of the alternative alloys are comparable to the alloy 2, indicating a similar precipitation sequence. Analysis of the alternative alloy microstructures as a function of the aging temperature demonstrated that the lower barriers to precipitation allowed the γ' phase to precipitate first. The γ'' precipitation follows but provides the greater contribution to the hardness. It was not clear if the γ'' phase was nucleated at γ' sites. Literature information confirmed this precipitation sequence (8,19).

The room temperature strength of Alloy 718 is very dependent on columbium contents (Figure 1). Additions of titanium for the reduce columbium improved the strength of alloy 4 to a level above this linear relationship. The decrease in strength of alloy 1a at temperatures above 540°C is the result of γ'' strength decrease and weakened grain boundaries. The boron addition of alloy 1b improved the strength and ductility at all temperatures by preventing the formation of low fracture energy phases at the grain boundaries. The strength of alloys 4 and 5, with increased titanium levels, did not decrease as rapidly as alloy 1a at temperatures above 540°C . This was the result of γ' precipitation supplementing the γ'' strengthening at these temperatures, and grain boundary regions with no low fracture energy phases (M_{23}C_6 and M_6C carbides). This indicated that anti-phase boundary and ordering mechanisms may have replaced coherency strains as the dominant strengthening mechanism at temperatures above 540°C .

The stress-rupture properties of the alloys were also sensitive to the precipitate type and grain boundary strength. At temperatures above 540°C , the stress-rupture strength of alloy 5 was equal or superior to alloy 1a, indicating reduced grain boundary creep. It would be inappropriate to credit the improved stress-rupture properties to increased γ' precipitation. Grain boundary stabilization through boron additions improved the stress-rupture properties of the standard alloy. The increased ductility of superalloys containing boron makes further alloying possible with elements that would sacrifice ductility for improved strength.

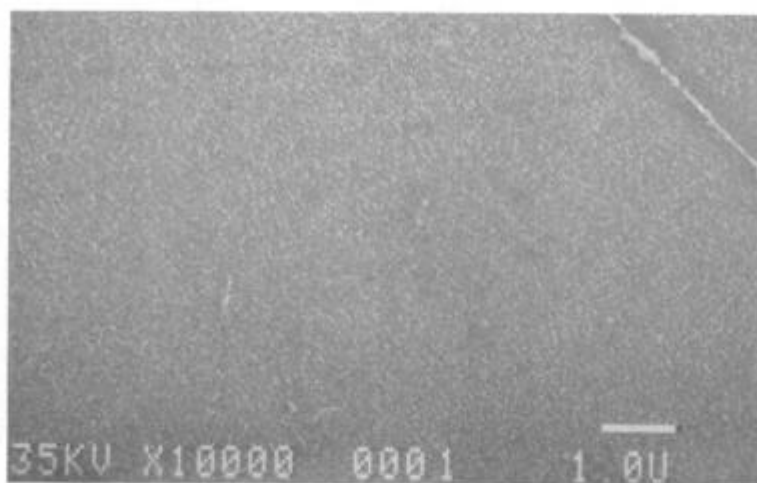


Figure 4 - Alloy 1a, 5.32Cb-0.87Ti-0.52Al;
 $(Al+Ti)/Cb = 0.66$, γ' precipitation.

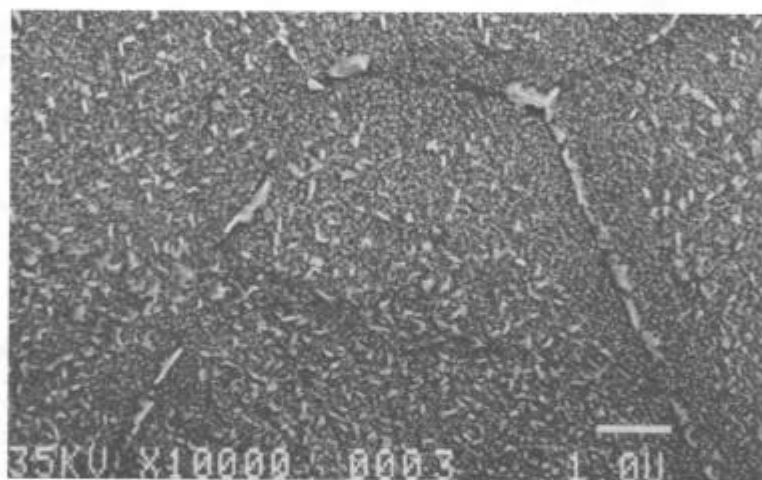


Figure 5 - Alloy 2a, 3.20Cb-0.87Ti-0.55Al;
 $(Al+Ti)/Cb = 1.14$, $\gamma'' + \gamma'$ precipitation.

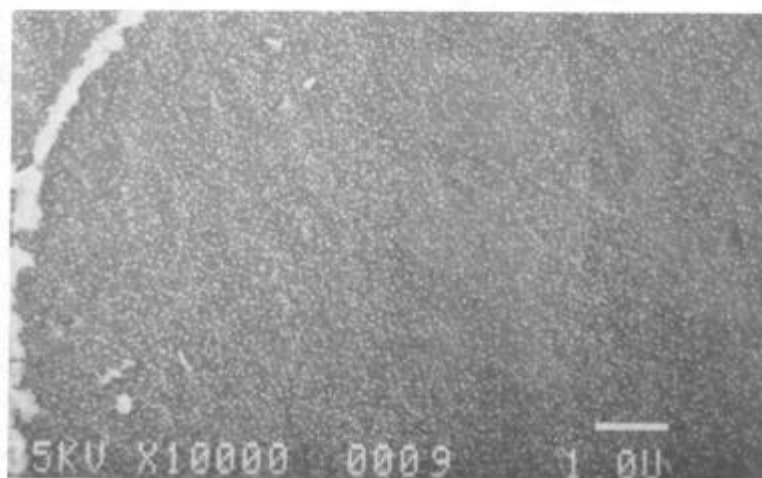


Figure 6 - Alloy 3a, 1.10Cb-0.87Ti-0.52Al;
 $(Al+Ti)/Cb = 3.36$, γ' precipitation.

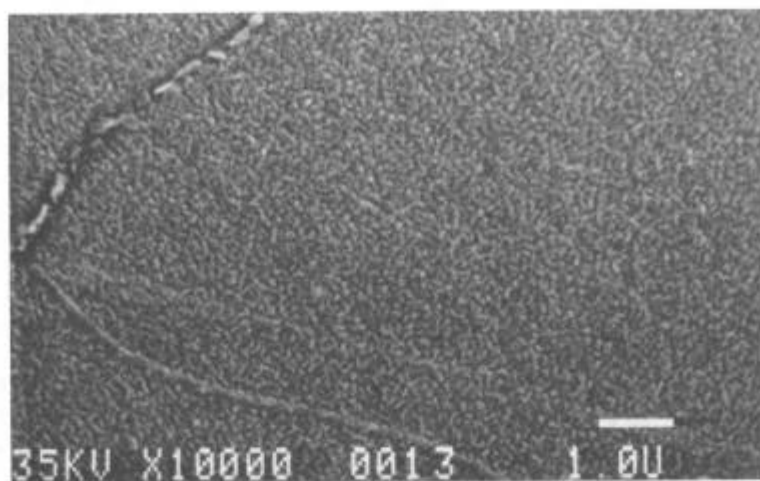


Figure 7 - Alloy 5, 3.89Cb-1.27Ti-0.55Al;
(Al+Ti)/Cb = 0.97, $\gamma'' + \gamma'$ precipitation;

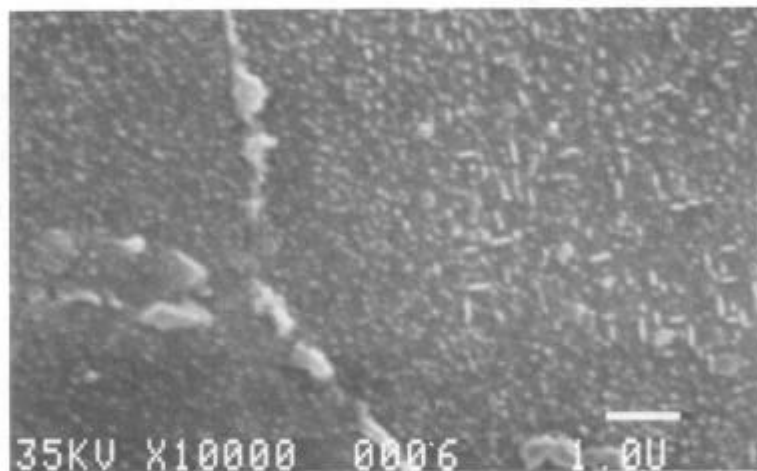


Figure 8 - Alloy 5, 3.89Cb-1.27-0.55Al.

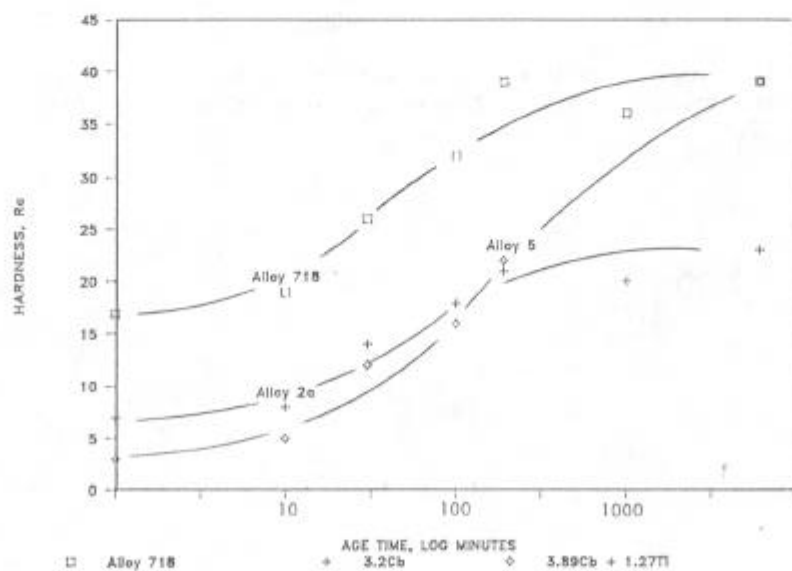


Figure 9 - Effect of aging time on alloy hardness.

Conclusions

Based on the results described above, the following conclusions can be made:

- 1) There is a linear relationship between the columbium content of Alloy 718 and the γ'' precipitation. The decrease γ'' precipitation results in a decrease in tensile strength at room temperature and 650°C.
- 2) Additions of solid solution strengthening elements for reduced columbium does not recover the properties of the original alloy. Substitution of γ'' forming elements will recover a large portion of the original Alloy 718 strength and stress-rupture properties. The (Ti+Al)/Cb ratio will determine the proportion of the strengthening phases that will form (γ'' vs. γ').
- 3) At temperatures above 540°C, the tensile properties of Alloy 718 decrease sharply because of lower grain boundary strength and coherency strain strengthening. However, with increased γ' formers such as titanium, the tensile properties decrease linearly between room temperature and 650°C. The stability of the γ' strengthening and cleaner grain boundaries, an alloy with 3.89Cb and 1.27Ti exhibited stress-rupture properties superior to Alloy 718 at 595 and 650°C.
- 4) The addition of boron to Alloy 718 produced a coincident increase in yield strength, tensile strength and ductility. The boron also greatly improved the stress-rupture life of the standard alloy. Additions of boron could be expected to also improve the tensile properties of reduced columbium alloys.

References

Due to space constraints, references will be available on request.

Acknowledgement

Funding for this program was provided by the National Aeronautics and Space Administration - Lewis Research Center, Cleveland, Ohio under grant NAG-3-268.