EFFECT OF VARYING ALUMINUM AND COLUMBIUM CONTENT ON HARDNESS AND TENSILE PROPERTIES OF 718 ALLOY HEAT TREATED FOR OIL FIELD REQUIREMENTS

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Abstract

Users of 718 for oil field applications generally require a minimum 0.2% R.T. tensile strength of 120 ksi. and hardness in the range of R_C 30- 40. The effect of compositional variations involving columbium and aluminum on hardness and tensile properties was evaluated using six different 718 compositions with columbium in the range 4.85 to 5.5 wt. percent and aluminum content of 0.37 to 0.69 wt. percent. The effect of solution and aging treatments on hardness was studied in the solution treatment range 1800° F to 1950° F /2h/ W.Q. Aging treatments of 4h and 8h in the range 1200° F to 1500° F were evaluated. Results show that heat treatments of 1875°F/2h/WQ + 1450° F/8h/AC and 1900° F/2h/WQ + 1425° F or 1450° F/8h/ AC result in a satisfactory combination of properties for all compositions evaluated.

Underaging appears to result in higher ductility for similar strength and hardness as compared to overaging but underaged properties are sensitive to variations in composition and aging temperature. The effect of variations in columbium and more specially aluminum are much reduced when higher solution temperatures and aging are used as compared to the 1750° F + 1325/1150° F age utilized for typical aerospace applications. Structures resulting are characterized.

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Introduction

Oil field users of 718 generally specify a minimum yield strength of 120 ksi., tensile strength of 150 ksi, minimum 20% elongation and 25% R.A. along with a hardness range of R_C 30-40. Reasons for this requirement are related to stress corrosion cracking resistance and are elaborated in other papers presented at this conference.

718 heat treated for aerospace applications generally utilizes a 1750° F solution treatment followed by a double age of 1325° F/8h/ cool 100° F per hour to 1150° F/ 8h/ AC. This treatment results in high strength and satisfactory stress rupture ductility. Previous research by Eiselstein and data published by Latrobe indicates that, when this type of treatment is used, increasing Al lowers tensile and yield strength without affecting elongation. Typical results are shown in Figure 1. It can be observed that the tensile strength of 718 is reduced from 180 ksi. to 160 ksi. by increasing aluminum content from 0.4 to 0.8%. Results plotted in Figure 1 indicate that the trend for lower strength with higher Al is observed for solution treatment temperatures as high as 1800° F.

The present study was conducted to define the effect of columbium and aluminum variations over a wider range of solution treatment and aging treatments. Oil field applications for 718 are generally at temperatures low enough that creep resistance is unimportant. Consequently, solution treatment temperatures in the range of 1800 to 1950° F and aging treatments for 4h and 8h in the range of 1200-1500° F were evaluated. Six different 718 alloy compositions having columbium contents of 4.85- 5.5 wt.% and aluminum contents of 0.37- 0.69% were VIM-VAR remelted in the laboratory and subsequently hot rolled to 1 .625" bar. The effect of solution and aging treatments on hardness was evaluated. Results were used to select heat treatments for tensile testing.

Procedure and Results

Two 420 lb. heats of vacuum induction melted 718 alloy

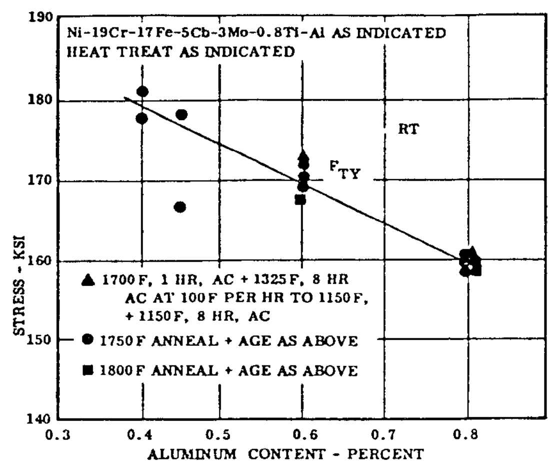

Figure 1 - Effect of Al on properties reported by Latrobe.

Table I. Modified 718 Compositions Weight Percent

	Heat 7795	Heat 7796	Heat 7797	Heat 7798	Heat 7799	Heat 7800
C	0.041	0.040	0.040	0.040	0.040	0.040
Mn	0.12	0.11	0.12	0.10	0.11	0.12
Si	0.11	0.12	0.12	0.13	0.13	0.14
P	0.008	0.006	0.007	0.002	0.002	0.002
S	0.003	0.003	0.003	0.003	0.003	0.003
Cr	18.32	18.22	18.26	18.41	18.47	18.43
Ni	52.80	52.84	52.82	52.59	52.95	52.75
Mo	3.10	3.10	3.13	3.08	3.10	3.11
Nb(*)	5.09	5.06	5.37	4.85	5.50	5.50
Al(*)	0.53	0.64	0.60	0.37	0.40	0.69
Ti	1.03	1.03	1.02	0.99	1.00	0.99
B	0.0037	0.0034	0.0035	0.0039	0.0038	0.0041
Fe(a)	18.6	18.6	18.3	19.2	18.1	18.0
Co	0.12	0.12	0.12	0.12	0.12	0.12
Cu	0.07	0.07	0.07	0.07	0.07	0.07

* = Recheck on C.E. Remelted Bar Stock
(all other values are pot analyses from electrodes).

(a) = Fe: Balance by subtraction.

were each cast into three electrodes. Aluminum and columbium additions were made to obtain the six different modifications shown in Table I. The VIM electrodes were vacuum arc remelted to 5" rd. ingots in the laboratory facilities. Ingots were homogenized at 2175° F for 64 hours, cooled to 2050° F and forged to 2.625" square billet. One 70 lb. billet was obtained from each ingot. The billet was prepared and rolled to 1.625" rd. bar from a rolling temperature of 2050° F.

Lengths about 5" long were cut from bar representing each composition for hardness studies. Pieces were solution treated 1800, 1850, 1875, 1900 and 1950°F/2h/ WQ. Following solution treatment the ends were trimmed and the lengths of bar were cut into discs which were subsequently quartered to pie shaped segments having cross sections representative of surface, mid-radius and near center. Quartered discs representing each heat and solution treatment were aged in the range 1200 to 1550 ° F (50°F intervals) for 4 and 8 hours followed by air cool. Hardness was determined on cross-section of each specimen. Results for the average of the near surface, mid-radius and near center hardness were measured and selected results are plotted in Figures 2 and 3. The as-hot rolled plus aged hardnesses were also characterized and results are plotted for comparison in Figure 4.

An aged hardness "band" is shown in Figures 2 through 4. Variation in composition results in this band having a width of about 5 Rockwell C points on average. Maximum hardness results from 1350° F aging and results are in general agreement with previous results. The maximum hardness obtained is greater than permitted by oil field specifications- indicating that underaging or overaging is required to meet property requirements. Underaging results in a steep hardness/ aging temperature curve as well as maximum variation in hardness resulting from composition differences. Overaging results in less hardness variation as a result of temperature or compositional variations and there is less variation in hardness with change in aging temperature. This greater predictability provided by overaging makes it preferable in production practices where some variation in composition and heat treatment can be anticipated.

Oil field specifications generally require that surface hardness be recorded to assure that the surface of the bar is within the specified hardness ranges. Surface hardness was investigated using pie shaped specimens solution treated 1850, 1875 and 1900° F and aged at 1450° F. Parallel flats were ground and surface hardness was sequentially measured at depths of 0.01", 0.03" and 0.06". The first two depths were measured in Rockwell A to minimize cold working deeper sections with the hardness indenter. The final step - 0.06" deep- was measured in Rockwell C. In practically all cases

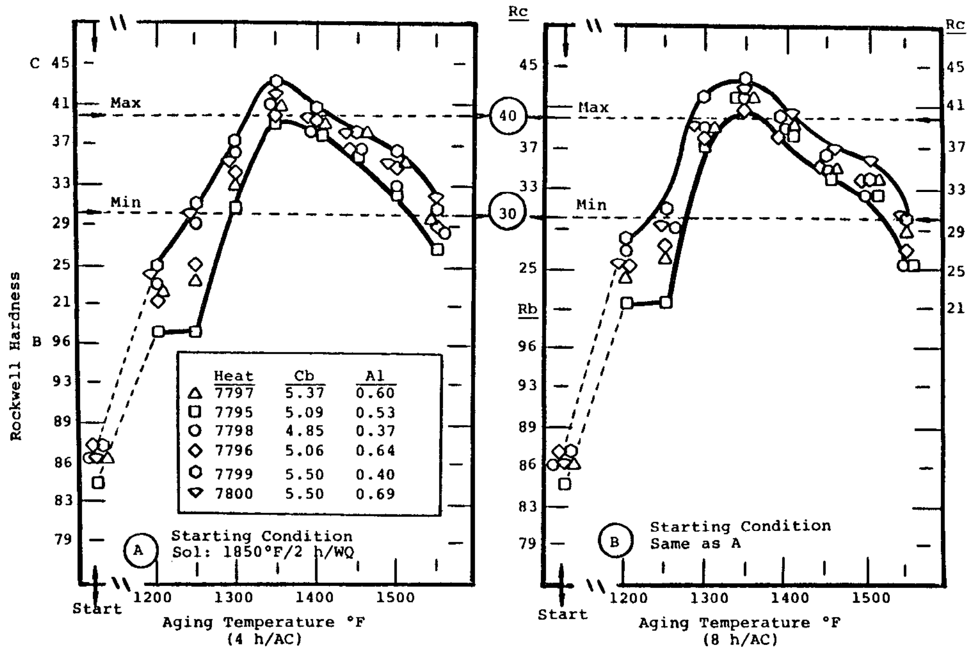

Figure 2 - Effect of aging temperature on the hardness of Modified 718 Alloy. Cross section, average -- 1-5/8" rd. -- Solutioned 1850°F/2 h/WQ.

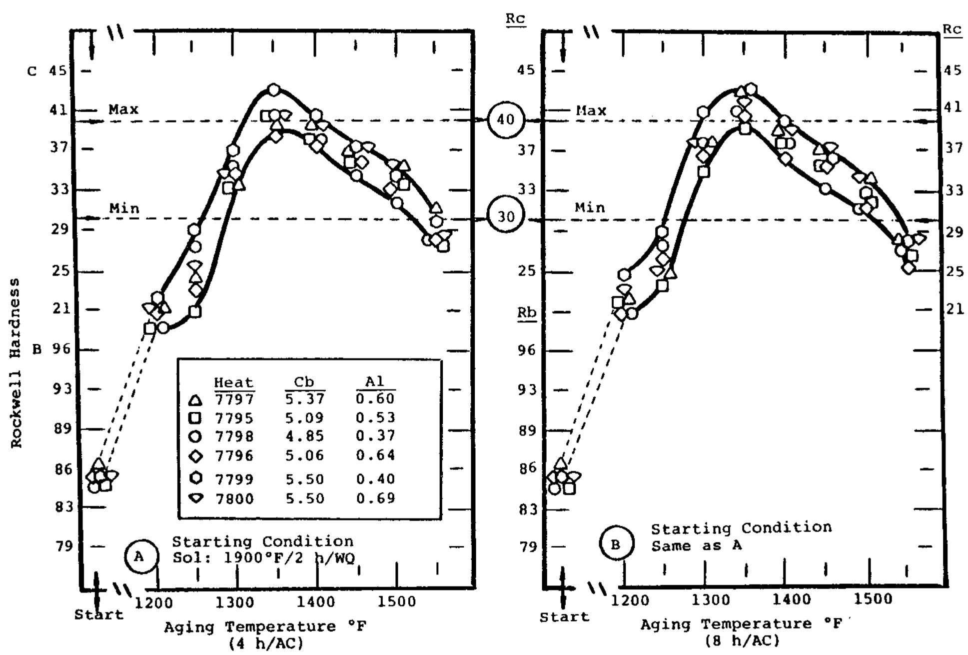

Figure 3 - Effect of aging temperature on the hardness of Modified 718 Alloy. Cross section, average -- 1-5/8" rd. -- Solutioned 1900°F/2 h/WQ.

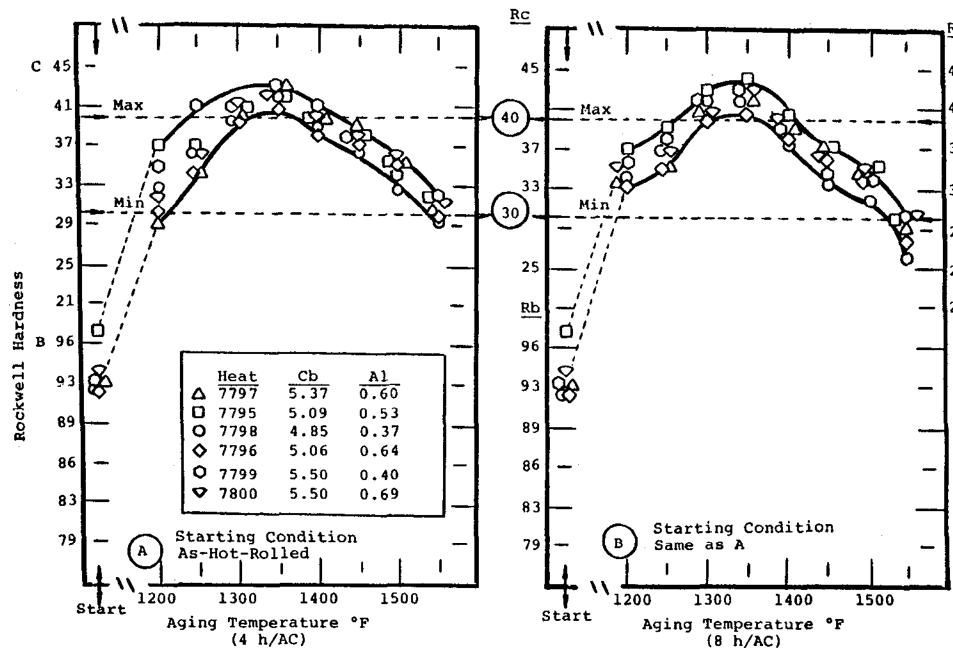

Figure 4 - Effect of aging temperature on the hardness of Modified 718 Alloy. Cross section, average -- 1-5/8" rd. -- As-hot-rolled

when the surface hardness measured was higher, it was within 0.5 R_C of the average value mentioned/plotted earlier. In a few cases, the surface hardness was up to 1 R_C higher and in only one case (Heat 7799- solution treated at 1875° F) it was 2 R_C higher. In general, it appears that cross sectional hardness values are an accurate reflection of near surface hardness.

Tensile properties were investigated for 1850° F and 1900° F solution treatments. Aging was 8h/AC at 1275° F (underaging) and 1425° F and 1450° F (overaging). One set of tensile test specimens was solution treated 1875° F/ 2h/ WQ plus age 1450° F/ 8h/ AC. Response to typical aerospace heat treatment was also tested. Results of tensile tests are shown in Tables II thru VII. Hardness measurements made on the threaded section are also indicated. Selected data are plotted in Figures 5 through 12. Duplicate tensile results were conducted and the values plotted represent the average. The horizontal axes are drawn to illustrate trends and are not to scale. Heats with Nb content of 5.09% and 5.06% are plotted as 5.1% Nb heats. Al contents are similarly rounded with the 0.64 % and 0.60 % Al heats plotted as 0.6% Al.

Figure 5 shows that use of the 1750° F + double age treatment specified for aerospace applications results in differences in yield strength as a function of composition. Decreased yield strength with increased Al was observed at various Nb levels evaluated. The effect of Al appears to be more acute at lower Nb content. Increasing Nb content increases the yield strength. Hardness using this treatment is

Table II. Room Temperature Tensile Test Results

Heat 7795 Cb 5.09 Al 0.53

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	174.1	203.4	21.5	46.9	43
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	105.5	156.6	43.6	58.3	28
2. 1425°F/8h/AC	133.4	179.9	27.6	52.3	37
3. 1450°F/8h/AC	124.9	175.8	27.9	52.7	36
1875°F/2h/WQ + Age 1450°F/8h/AC	123.6	174.5	28.1	51.4	35
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	101.9	152.4	46.4	60.4	26
2. 1425°F/8h/AC	134.5	179.5	27.2	51.5	36
3. 1450°F/8h/AC	127.5	177.0	27.0	50.4	35

Table III. Room Temperature Tensile Test Results

Heat 7796 Cb 5.06 Al 0.64

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	164.5	193.1	23.0	48.3	42
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	112.9	164.1	40.3	58.9	33
2. 1425°F/8h/AC	138.8	184.2	25.7	50.3	38
3. 1450°F/8h/AC	130.8	180.6	26.0	49.1	36
1875°F/2h/WQ + Age 1450°F/8h/AC	132.3	181.6	27.3	50.7	37
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	96.9	146.6	47.1	57.3	25
2. 1425°F/8h/AC	131.8	176.4	28.3	49.4	37
3. 1450°F/8h/AC	126.0	174.4	27.8	48.2	35

Table IV. Room Temperature Tensile Test Results

Heat 7797 Cb 5.37 Al 0.60

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	170.1	196.3	20.5	46.0	42
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	118.0	166.4	39.7	58.9	32
2. 1425°F/8h/AC	142.5	184.9	25.5	49.1	39
3. 1450°F/8h/AC	136.2	183.2	25.2	48.4	37
1875°F/2h/WQ + Age 1450°F/8h/AC	136.0	183.7	26.5	47.7	38
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	103.5	150.4	45.5	57.7	27
2. 1425°F/8h/AC	137.4	179.8	27.6	47.6	38
3. 1450°F/8h/AC	130.8	178.9	27.6	45.5	37

Table V. Room Temperature Tensile Test Results

Heat 7798 Cb 4.85 Al 0.37

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	174.2	200.1	20.8	47.2	43
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	117.9	167.2	38.4	58.0	34
2. 1425°F/8h/AC	135.6	182.0	26.9	48.5	37
3. 1450°F/8h/AC	126.7	174.6	25.5	50.9	36
1875°F/2h/WQ + Age 1450°F/8h/AC	127.9	174.4	27.3	52.3	35
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	103.3	153.1	44.1	58.5	27
2. 1425°F/8h/AC	130.3	175.0	26.0	49.0	36
3. 1450°F/8h/AC	122.3	169.9	27.8	49.8	34

Table VI. Room Temperature Tensile Test Results

Heat 7799 Cb 5.5 Al 0.4

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	182.5	204.8	16.6	40.7	44
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	128.3	175.6	34.1	57.4	35
2. 1425°F/8h/AC	145.2	187.4	23.7	46.1	39
3. 1450°F/8h/AC	135.9	181.5	25.1	48.1	38
1875°F/2h/WQ + Age 1450°F/8h/AC	136.3	182.6	25.2	47.7	37
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	122.7	170.1	37.6	57.0	34
2. 1425°F/8h/AC	143.8	186.5	23.9	46.5	39
3. 1450°F/8h/AC	134.2	180.1	24.5	46.8	37

Table VII. Room Temperature Tensile Test Results

Heat 7800 Cb 5.5 Al 0.69

	0.2% Y.S. (ksi)	U.T.S. (ksi)	Elong. %	R.A. %	Average Hardness (Rc)
1750°F/2h/WQ + 1325°F/8h/ Cool 100°F/h to 1150°F/8h/AC	172.1	197.3	21	44.9	42
1850°F/2h/WQ + Age					
1. 1275°F/8h/AC	120.5	168.5	39.1	58.3	33
2. 1425°F/8h/AC	144.5	189.4	25.1	48.6	39
3. 1450°F/8h/AC	136.1	185.1	26.0	48.9	38
1875°F/2h/WQ + Age 1450°F/8h/AC	138.3	186.3	25.3	47.2	38
1900°F/2h/WQ + Age					
1. 1275°F/8h/AC	101.5	149.9	47.3	57.0	28
2. 1425°F/8h/AC	139.9	183.1	26.6	47.8	38
3. 1450°F/8h/AC	134.3	182.0	25.9	47.0	37

Figure 5

EFFECT OF NB AND AL ON ROOM TEMP TENSILE

1750 F/2H/WQ + 1325/8H/COOL 100 F/H TO 1150 F/8H/AC

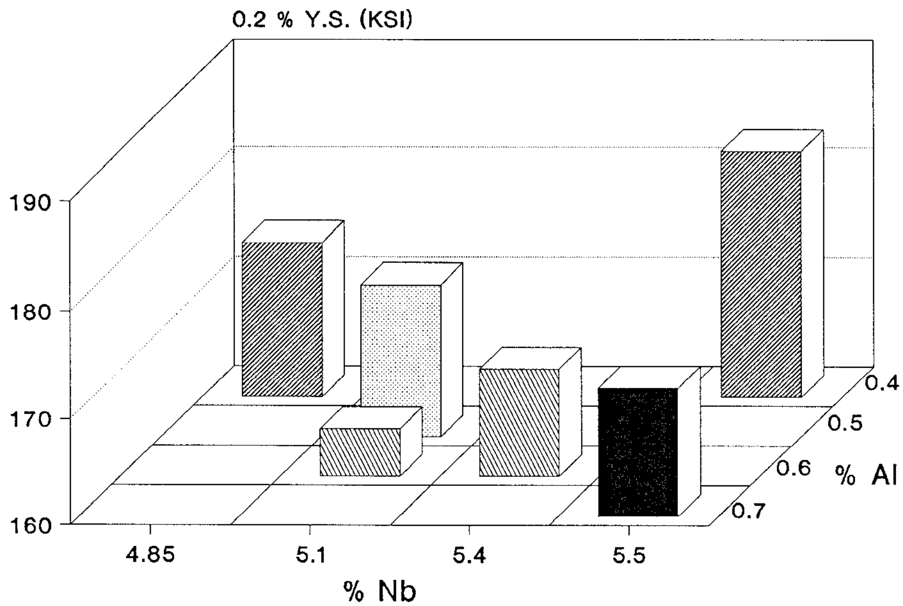

Figure 6

EFFECT OF NB AND AL ON ROCKWELL HARDNESS

1750/2H/WQ +1325/8H/COOL 100 F/H TO 1150/8H/AC

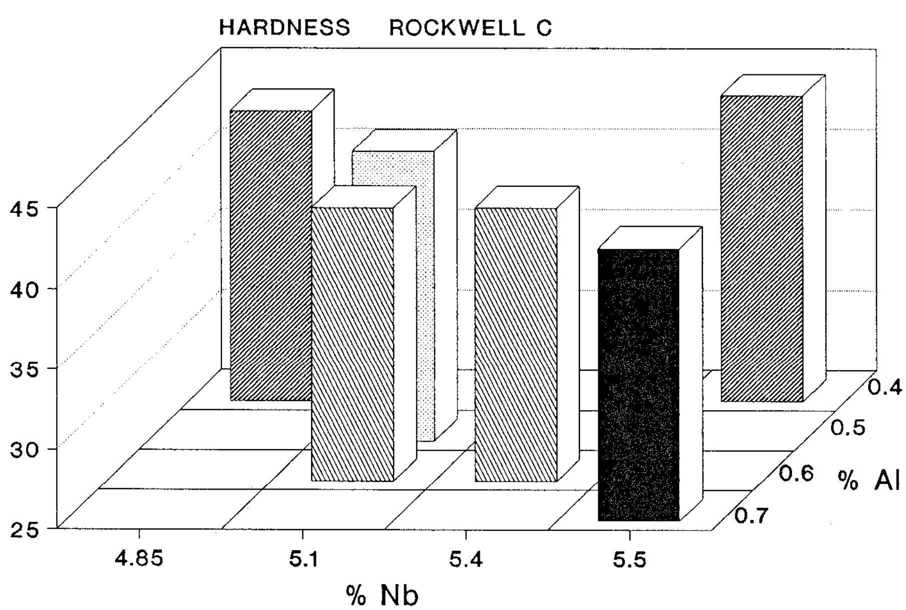

Figure 7

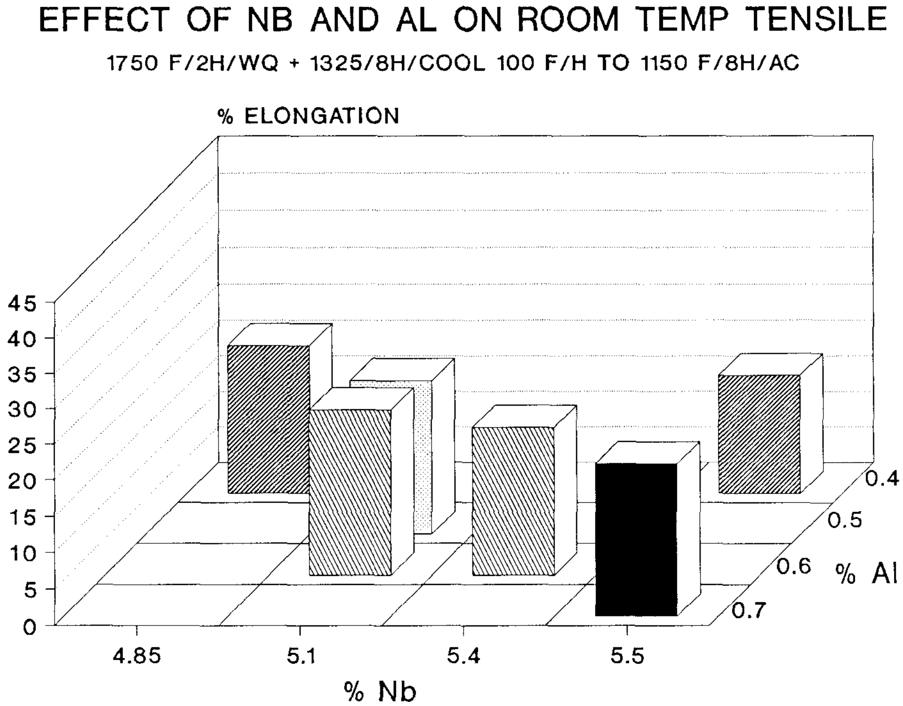

Figure 8

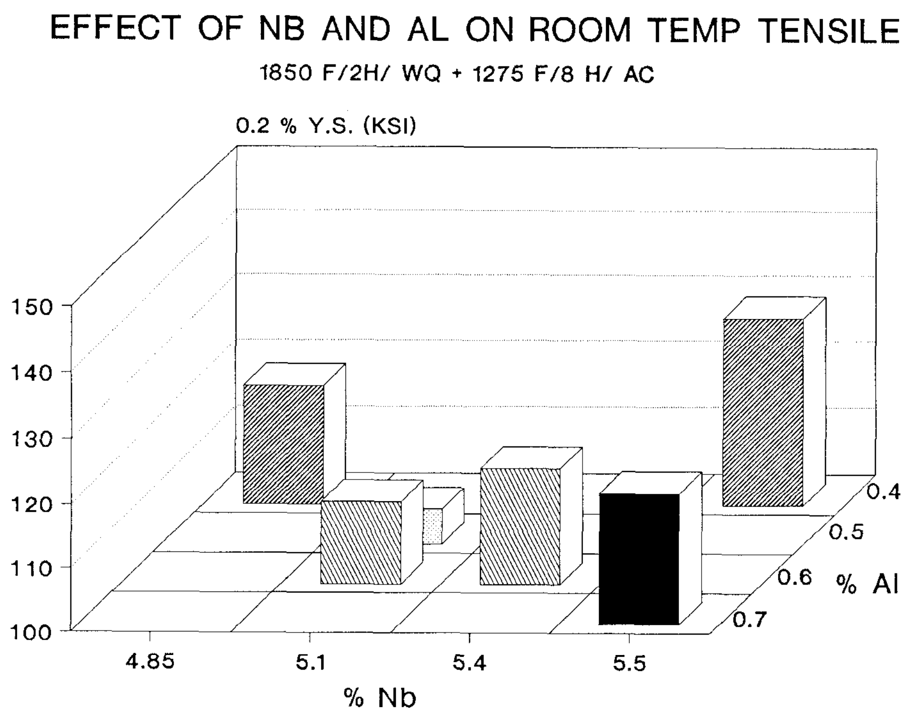

Figure 9

EFFECT OF NB AND AL ON ROOM TEMP TENSILE

1850 F/2H/WQ + 1275/8H/AC

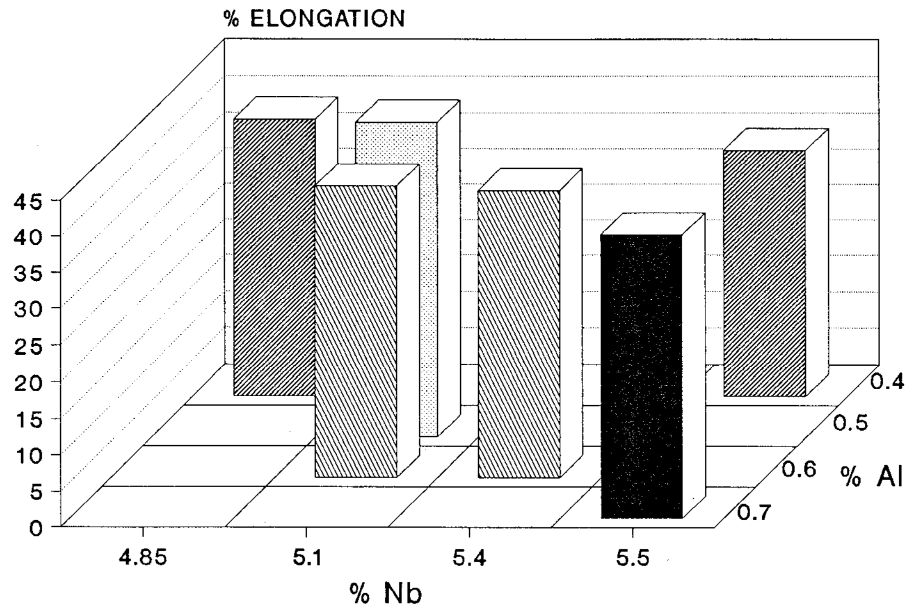

Figure 10

EFFECT OF NB AND AL ON ROOM TEMP TENSILE

1875 F/2H/ WQ + 1450 F/8 H/ AC

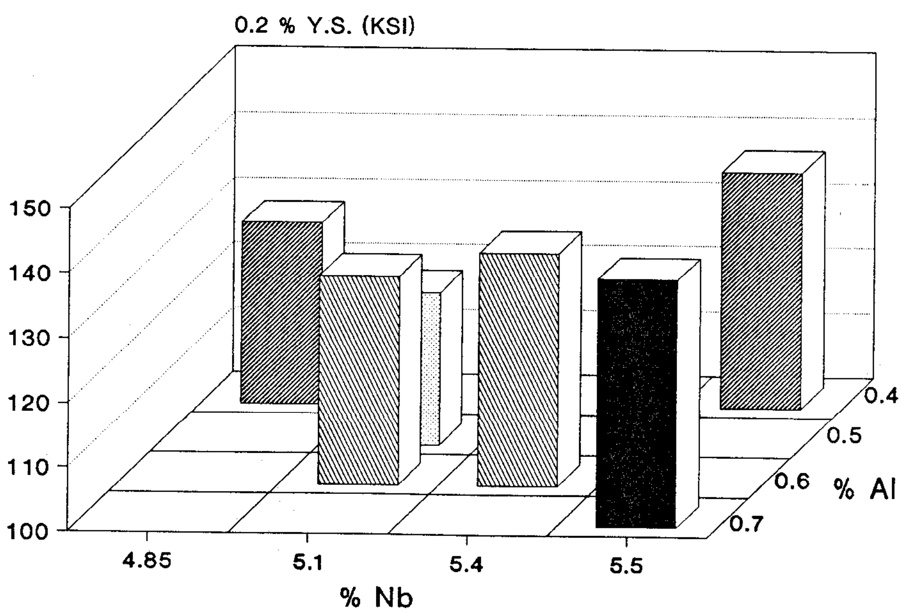

Figure 11

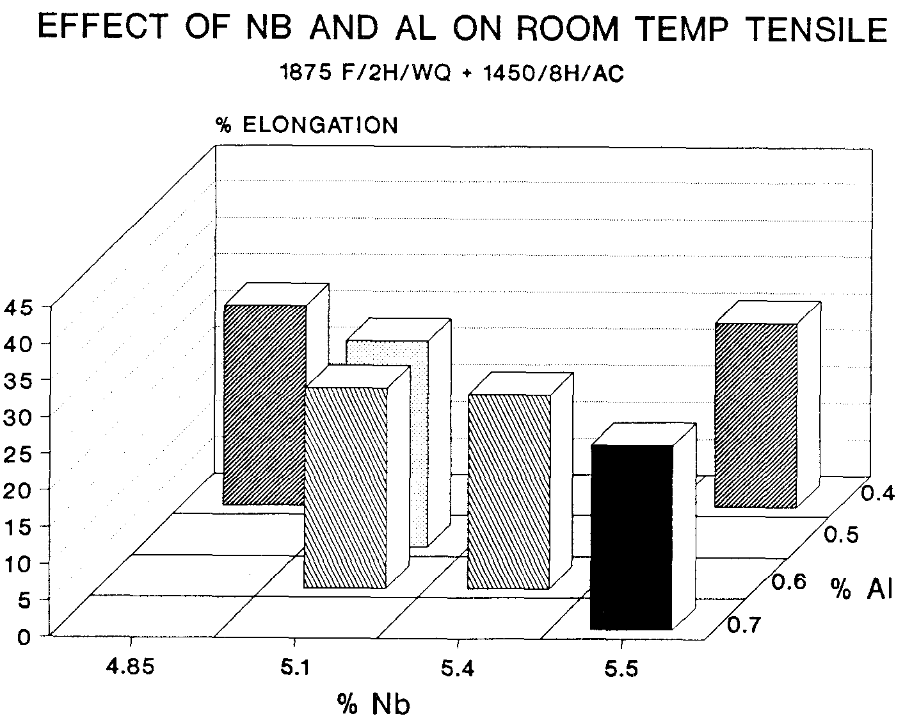

TABLE VIII[3]

X-RAY DIFFRACTION AND EDAX ANALYSIS

Sample		Phases	w/o Al in Residue
1850°F/2h/WQ	7797 (5.37 Nb, 0.6 Al)	fcc	8.92
+1275°F/8h/AC	7798 (4.85 Nb, 0.37 Al)	fcc	4.1
	7799 (5.5 Nb, 0.4 Al)	fcc	----
1850°F/2h/WQ	7797 (5.37 Nb, 0.6 Al)	fcc (bct)	4.61
+1450°F/8h/AC	7798 (4.85 Nb, 0.37 Al)	bct, fcc	2.69
	7799 (5.5 Nb, 0.4 Al)	bct, fcc	1.96
1850°F/2h/WQ	7797 (5.37 Nb, 0.6 Al)	fcc	8.11
+1350°F/8h/AC	7798 (4.85 Nb, 0.37 Al)	fcc	3.55
	7799 (5.5 Nb, 0.4 Al)	fcc	----

much higher than acceptable (Figure 6) but appears to follow trends similar to the yield strength. A minor degradation in ductility accompanies the higher strength obtained in low Al, high Nb heats (Figure 7).

Use of 1850°F/1900° F solution treatments with a 1275° F age retains the trend for higher strength obtained in higher Nb heats (Figure 8). Lower Al still results in a higher strength but the effect is reduced compared to 1750° F solution treatment when the 1850° F solution treatment is used. Trends are more acute for the 1900° F solution treatment. Due to the experimental design used, this effect is most apparent when comparing yield strength with varying Nb contents at constant Al contents of 0.4% and 0.6%. Data for the 5.1% Nb, 0.5% Al heat does not fit the trend observed. Very high tensile ductility is obtained via use of the 1275° F age with some reduction in elongation with higher Nb content (Figure 9). Elongation appears to be slightly higher for the low Nb, high Al compositions.

Use of high (1850/1900° F) solution temperatures combined with overaging (1425/1450° F) results in higher strength/hardness for higher Nb compositions but minimal decrease in strength with increasing Al is observed (Figure 10). Further, the elongation (Figure 11) and hardness of compositions evaluated also do not appear to be strongly dependent on Al content when a high solution treat/high age treatment is employed. Properties for all heats so heat treated meet the requirements of 120 y.s. minimum and hardness between R_C 30-40. For all compositions evaluated, the property ranges were Y.S. - 122.3 ksi. to 138.3 ksi., U.T.S.- 169.9 to 186.3 ksi., Elongation- 24.5 to 28.1%, R.A. - 45.5 to 52.3% and hardness- 34 to 38 Rockwell C. This appears to be the preferred heat treatment if latitude in composition is desired. In general, use of the 1275° F aging was judged to result in unacceptably low yield strength. Trends observed indicate that underaging results in higher ductility for similar strength compared to overage properties.

The microstructure of selected samples was characterized using scanning electron microscopy as well as x-ray diffraction and EDAX analysis of extracted residue by Rogers and Burns. Scanning electron microscopy indicated that the precipitate in specimens heat treated using the aerospace treatment was 300 Å in size. No precipitate could be resolved in any of the specimens aged at 1275° F at magnifications upto 30,000 X (Figure 12 A). Additional aging of 1275° F/20 h resulted in resolvable precipitates at 30,000 X (Figure 12 B). Photomicrographs of two heats with fairly diverse compositions - Heat 7797 (5.37% Nb, 0.6% Al) and Heat 7798 (4.85% Nb, 0.37% Al) that were aged at 1450° F following an 1850° F solution treatment are shown in Figures 13 and 14. There is not much difference in the appearance of the two microstructures. The grain boundaries do not show precipitation to a degree that can degrade corrosion or ductility. Morphology did not firmly establish the phase identity of the aging precipitate but results of previous studies would indicate that the precipitate is γ''.

Table VIII shows results of X-ray diffraction and EDAX analysis of extracted residue on selected specimens. 1% ammonium sulfate and citric acid was the extracting solution. The extraction was performed at 5 V for 2h. X-ray diffraction was performed using monochromated Cu radiation. These results suggest that aging at 1275° F and 1350° F results in the formation of fcc γ'. γ''precipitation was observed in samples aged at 1450° F. The fcc γ' is known to contain a higher Al content and the results shown agree with previous work. Higher Al content was observed in residue from heats containing higher Al. Also, in agreement with the work of Collier et. al and Cozar and Pineau the results on the 1450°F aged specimens indicate that higher Al/Ti and (Al+Ti)/Nb ratio result in 718 that is less prone to overaging and favors formation of γ' compared to γ''. It has been shown that increasing Al/Ti and (Al+Ti)/Nb ratios results in a finer sized precipitate and a more uniform distribution of precipitates.

Conclusions

1. The requirements of 120 ksi. minimum yield strength and 30- 40 R_C hardness was met for all compositions evaluated

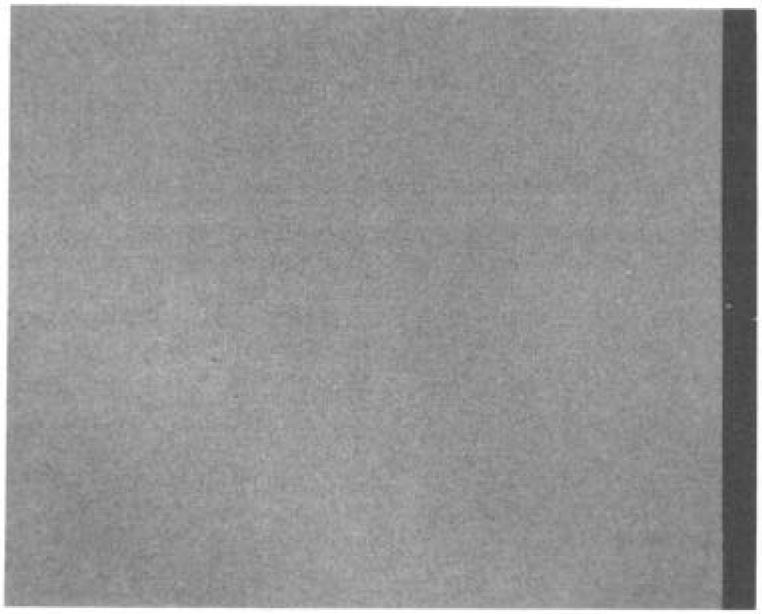

Figure 12A - 30,000X - SEM Micrograph of
Heat 7799 (5.5 Nb, 0.4 Al) treated
1850°F/2h/WQ + 1275°F/8h/AC

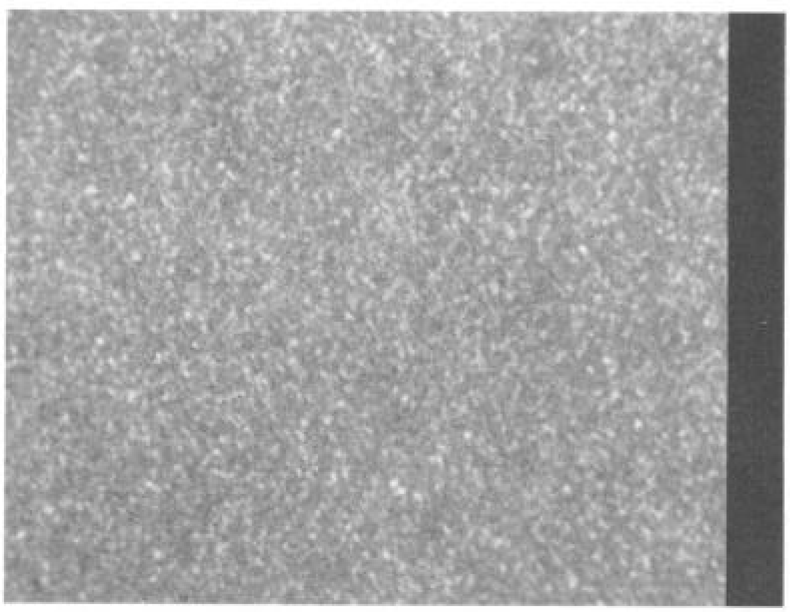

Figure 12B - 30,000X - SEM Micrograph of
Heat 7799 (5.5 Nb, 0.4 Al) treated
1850°F/2h/WQ + 1275°F/8h/AC + 1275°F/20h

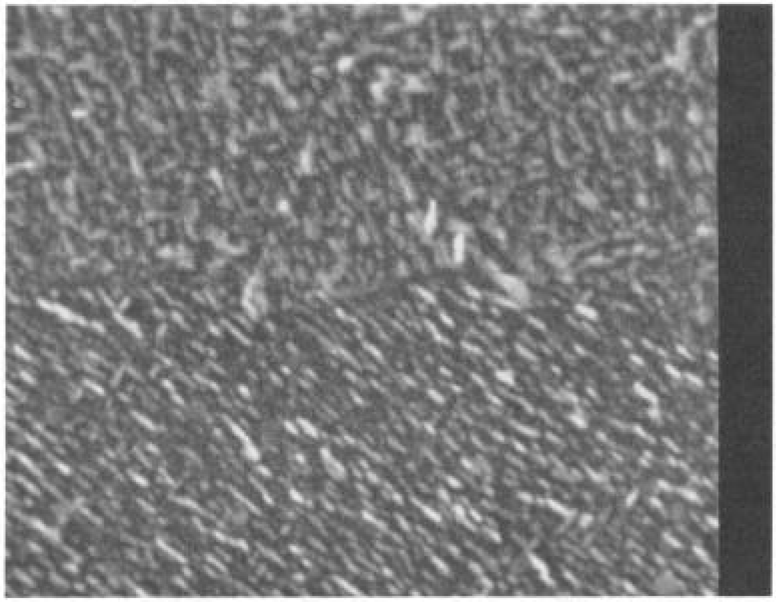

Figure 13 - 30,000X - SEM Micrograph of Heat 7797 (5.37 Nb, 0.6 Al) treated 1850°F/2h/WQ + 1450°F/8h/AC

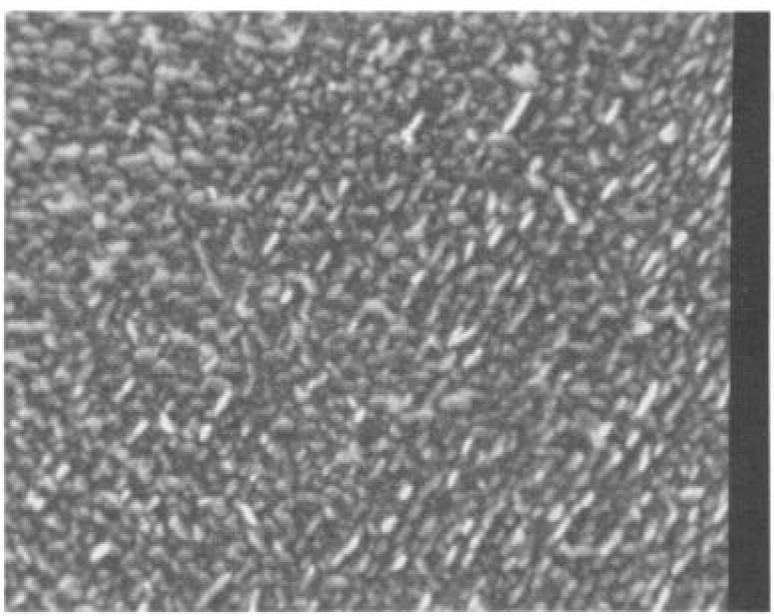

Figure 14 - 30,000X - SEM Micrograph of Heat 7798 (4.85 Nb, 0.37 Al) treated 1850°F/2h/WQ + 1450°F/8h/AC

when heat treated 1875°F/2h/WQ + 1450°F/8h/AC or 1900°F/2h/WQ + 1425°F or 1450° F/8h/ AC.

2. Underaging as compared to overaging appears to improve tensile ductility for similar strength and hardness but is sensitive to minor variations in composition and aging temperature.

3. The effects of Nb and Al content are dependent on the heat treatment employed. Increased Nb and decreased Al increase strength for aerospace type treatments. However, use of higher solution temperatures and underaging (1275° F) reduces this dependence. Use of high (1850/1900° F) solution temperatures combined with overaging (1425/1450° F) specified for oil field applications results in higher strength/hardness for higher Nb compositions but no decrease in strength with increasing Al is observed.