

EFFECT OF NICKEL CONTENT ON DELTA SOLVUS TEMPERATURE AND MECHANICAL PROPERTIES OF ALLOY 718

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Abstract

Knowledge and control of the delta phase (Ni_3Nb) solvus temperature is critical to optimize processing and heat treatment of alloy 718 for best mechanical properties. This is the case in aerospace applications where delta phase is used to produce fine grain structures during forging, and in oil-field applications where the delta phase is dissolved during solution treatment. Delta phase solvus is primarily controlled by niobium content. The effect of nickel content on delta phase solvus is not well understood as thermodynamic models and previous experiments have produced conflicting results. The purpose of this study was to confirm the effect of nickel content on delta solvus temperature using experimental heats and to document mechanical properties vs. nickel content. In addition, solvus results were compared with modeling predictions and an improved database was developed to more accurately predict delta phase solvus temperature in alloy 718.

Introduction

Delta is an orthorhombic phase that is incoherent with the face-centered cubic matrix. Delta phase size, morphology, and distribution dictate whether the phase is beneficial or detrimental to alloy performance. A uniformly-dispersed globular precipitate will facilitate grain refinement while a lathlike or needle morphology will adversely affect stress rupture and fracture toughness properties [1,2].

For aerospace applications, knowledge of the delta solvus temperature is essential for identifying forging and heating temperature practices that incorporate a dispersed delta phase to obtain the desired uniform fine grain structures. For oil-field applications where delta phase is undesirable for fracture toughness, knowledge and control of the solvus temperature is important for determination of super-solvus solution treatment temperatures to fully dissolve delta phase while limiting grain growth.

It is well known that the delta phase solvus is primarily controlled by niobium content [3]; however, niobium content in alloy 718 is typically tightly controlled by industry specifications, either directly or indirectly by the mechanical properties required (niobium controls strength). The effect of nickel content on delta phase solvus is not well understood and thermodynamic models and previous experiments have produced conflicting results. Work by E. Raymond [4] indicates that delta solvus of 718 billet increases with nickel content in the 52-54% range. Other researchers [5] found no significant effect of nickel, cobalt, and iron levels on delta solvus temperature. Modeling simulations using the databases from Thermotech and CompuTherm

predict conflicting trends for nickel. Also, both databases predict solvus temperatures significantly higher than the 990-1015°C range typically observed in alloy 718 [3,6].

Computational thermodynamic modeling based on the CALPHAD (CALculation of PHase Diagrams) approach can be employed to predict the fractions of equilibrium and metastable phases, and their compositions in multi-element systems. Modeling is now routinely used in the metals industry to better understand the physical metallurgy of existing alloys and to facilitate the development of new alloys. While commercially available thermodynamic modeling software, such as Thermo-Calc, Pandat®, and JMatPro are becoming increasingly user-friendly, the key to using these predictions effectively is the accuracy of the databases being used. It is important for the users to understand the strengths and limitations of each database so that they can apply the calculation results appropriately to the alloy design and evaluation process.

Currently, there are two major commercial databases available for nickel-base superalloys, Ni-DATA from Thermotech [7] and PanNickel from CompuTherm [8]. Zhao et al. evaluated the Ni-DATA database by comparing the thermodynamic predictions with experimental observations [9]. Zhao's work showed that the thermodynamic calculations predict the stability of the gamma prime phase reasonably well but the predictions for TCP phases are less satisfactory. A recent paper by Olson et al. indicated that Ni-DATA (version 7) is more favorable compared to PanNickel, because it predicts the volume fraction and composition of gamma prime phase accurately [10]. However, systematic assessment of the databases for delta phase predictions has not been performed.

The objectives of this study were: 1.) to confirm the effect of nickel content on delta solvus temperature using experimental heats; 2.) to document effects on room temperature tensile, Charpy V-notch impact, and stress-rupture properties; and 3.) to assess the thermodynamic databases for delta phase prediction in alloy 718. Niobium variations were also evaluated for comparison.

Experimental Procedure

Alloy Modeling

The effects of nickel and niobium contents on delta phase solvus temperature of alloy 718 were studied using the commercial thermodynamic modeling softwares Thermo-Calc and Pandat. Two databases were employed in this work, Ni-DATA, version 8 (TTNi8) and PanNickel.

Experimental Alloy Processing

Three nickel levels, within the typical 50-55% broad range for alloy 718, were selected for evaluation (51.5%, 53%, and 54.5%). All compositions are expressed in weight percent. Niobium levels of 5.0%, 5.2%, and 5.4% were also evaluated at the intermediate 53% nickel level. Five 40-lb (18-kg) laboratory heats were vacuum-induction melted and cast as 100-mm square ingots. The chemical compositions of the heats are listed in Table I.

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Table I. Chemical Compositions (Wt%) of Alloy 718 Experimental Heats

Heat	C	Mn	Si	P	S	Ni	Nb	Cr	Mo	Ti	Al	B	Fe
1	0.03	0.07	0.10	0.006	6 ppm	51.53	5.17	18.26	2.97	0.98	0.50	0.004	20.2
2	0.03	0.07	0.10	0.006	<5 ppm	53.05	5.00	18.24	2.97	0.98	0.51	0.004	18.9
3	0.03	0.07	0.10	0.006	6 ppm	53.08	5.18	18.29	2.97	0.98	0.50	0.004	18.7
4	0.03	0.07	0.10	0.006	6 ppm	53.04	5.42	18.24	2.97	0.98	0.50	0.004	18.5
5	0.03	0.07	0.10	0.006	<5 ppm	54.52	5.18	18.26	2.97	0.98	0.50	0.004	17.3

The ingots were homogenized, soaked at 1120°C (2050°F), and press-forged to 64-mm double-octagon bars. After bar turning, the bars were then hot-rolled to 19-mm round bars from 1120°C (2050°F) using hand rolling mills.

To compare response to solution treatment, as-rolled bar samples were treated at temperatures ranging from 925°C to 1025°C (1700-1875°F) in increments of 28°C (50°F) for one hour and water quenched. Smaller temperature increments were used in the temperature ranges where recrystallization and delta solution occurred. Hardness and microstructure of longitudinal mid-radius sections of the solution-treated samples were evaluated. Metallographic specimens were ground and polished using conventional preparation techniques. Samples were swabbed with aqua regia etchant to reveal grain structure and secondary phases.

Solvus and Phase Analysis

Calorimetry was utilized to evaluate melting range and solvus temperatures of the alloys. Disks (40 mg) were electric discharge machined from as-forged and heat-treated samples. A TA Instruments® model Q600 differential thermal analyzer (DTA) was used in this study. Alumina crucibles, argon cover gas with a 100 mL/min flow rate, and 20°C/min heating rate were applied to all scans.

The gamma-double-prime (γ'') hardener phase solvus was measured using DTA and the solvus temperature corresponds to the peak maximum of the endothermic peak. Metallographic assessment of the delta solvus was performed in place of DTA because the endothermic peaks associated with dissolution of the delta phase are difficult to resolve. The poor resolution is attributed to slow reaction kinetics [11].

Transmission electron microscopy was incorporated for quantification of compositions and verification of phases. Carbon extraction replica specimens were prepared from 925°C (1700°F)/1hour treated samples from heats #1 and #3, which represent the low-nickel and intermediate-nickel contents. A Philips model EM-420T operating at 120kV provided images, energy dispersive spectrums (EDS), and selected area electron diffraction patterns (SAED) to identify the minor phases.

Mechanical Property Evaluation

For evaluation of mechanical properties, solution and aging heat treatments commonly used for aerospace and oil-field 718 products were applied. The heat treatments were as follows:

Aerospace: 954°C (1750°F)/1h/AC + 718°C (1325°F)/8h/FC to 621°C (1150°F)/8h/AC

Oil-Field: 1024°C (1875°F)/1h/OQ + 785°C (1445°F)/6h/AC

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Heat-treated blanks were machined/ground to 6.35-mm gage diameter tensile and stress-rupture specimens and standard Charpy V-notch impact specimens (10-mm square). Hardness, tensile and impact tests were conducted at room temperature. Stress-rupture tests were conducted at a temperature of 649°C (1200°F) using a load of 758 MPa (110 ksi). Since stress-rupture testing is only relevant for applications above about 538°C (1000°F), only the aerospace-treated samples were evaluated in this test.

Results and Discussion

Phase Solvus and Microstructure

The microstructures of the hot-rolled bars were consistent among the five heats. A fine ASTM 11-12 grain size was obtained in all cases. The melting range was found to be 1260-1357°C (2300-2475°F). There were no significant differences in melting range within the 51.5-54.5% nickel and 5.0-5.4% niobium composition ranges.

Table II contains the solvus temperature data. Figure 1 shows that nickel content had little or no effect on gamma-double-prime (γ'') or delta solvus temperatures. Moreover, both solvus temperatures were found to be positively correlated with niobium content as would be expected. The delta solvus temperatures are similar to those documented by others [3,6]. The γ'' solvus was observed over the range of 915-926°C (1679-1699°F). Previous DTA investigations have measured γ'' solvus temperatures of 894°C (1641°F) [11] and 877°C (1611°F) [12]. Differences may be related to techniques used to evaluate the thermograms.

Table II. Solvus Temperatures of Alloy 718 Experimental Heats

Nickel (wt%)	Niobium (wt%)	Gamma Double Prime ¹		Delta Phase ²	
		°C	°F	°C	°F
51.5	5.2	921	1689	1002	1835
53.1	5.0	915	1680	993	1820
53.1	5.2	922	1691	1004	1840
53.0	5.4	927	1700	1010	1850
54.5	5.2	924	1695	1004	1840

1 – determined using DTA

2 – determined using metallography of treated samples

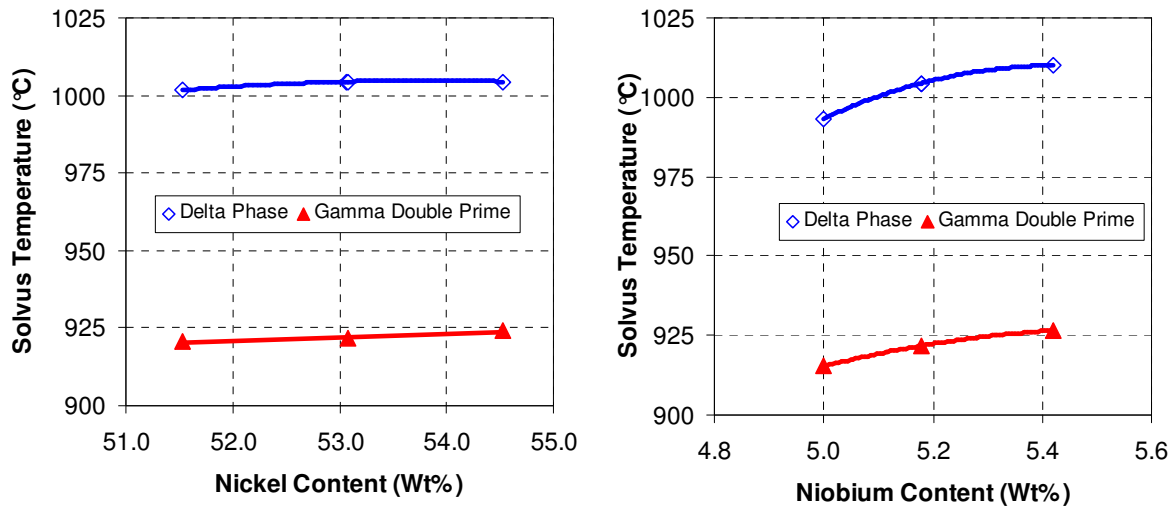


Figure 1. Effect of nickel and niobium contents on solvus temperatures of alloy 718 (determined experimentally using laboratory heats)

No grain growth was observed in specimens that were heat-treated below the delta solvus. The amount of delta phase was greater in samples with higher niobium content. Near the delta solvus temperature, the fraction of delta precipitates on grain boundaries was insufficient and grain coarsening was observed; the resultant microstructures exhibited mixed or duplex grain sizes.

Table III. Alloy 718 Phase Composition Data* (Wt%)

Heat	Phase	Ni	Fe	Cr	Nb	Ti	Al	Mo	Si
#1 (51.5%Ni, 5.2%Nb)	Delta	64.2	2.6	0.8	28.8	2.1	0.1	0.9	0.2
"	Laves	28.8	15.5	13.1	26.5	0.2	0.3	13.0	1.3
#3 (53.1%Ni, 5.2%Nb)	Delta	64.7	2.8	0.7	27.5	2.0	0.2	1.0	0.9
"	Laves	No Laves detected in this composition							

* - Data obtained by EDS of 925°C treated samples

Table III lists the compositions of the phases found in heats #1 and #3 after treatment at 925°C (1700°F) for 1 hour. Delta phase was confirmed (TEM/SAED) in these heats but was observed in all compositions heat treated below the delta solvus range of 993-1010°C (1820-1850°F). In the low-nickel heat, a few of the precipitates matched the Laves phase crystal structure and exhibited a composition significantly different from the matrix and delta precipitates. Extensive research has addressed the presence of Laves phase in alloy 718 in the as-cast condition [2,13,14]. The use of ingot homogenization treatments has been shown to dissolve the Laves phase and reduce the segregation of niobium in alloy 718. Likewise, control of niobium and silicon levels decreases propensity to form Laves. For these reasons, Laves phase is not common in wrought products of commercial alloy 718 in the solution-treated condition. Given that Laves phase was found in heat #1 and not in heat #3, this work confirms that a minimum critical nickel content is necessary to achieve a Laves-free microstructure in homogenized wrought products of alloy 718.

Modeling of Solvus Temperature

For both Ni-DATA, version 8 (TTNi8) and PanNickel databases, the predicted solvus temperatures are higher than our measurements as shown in Figure 2, but the predictions from PanNickel show smaller differences compared to the experimentally determined values.

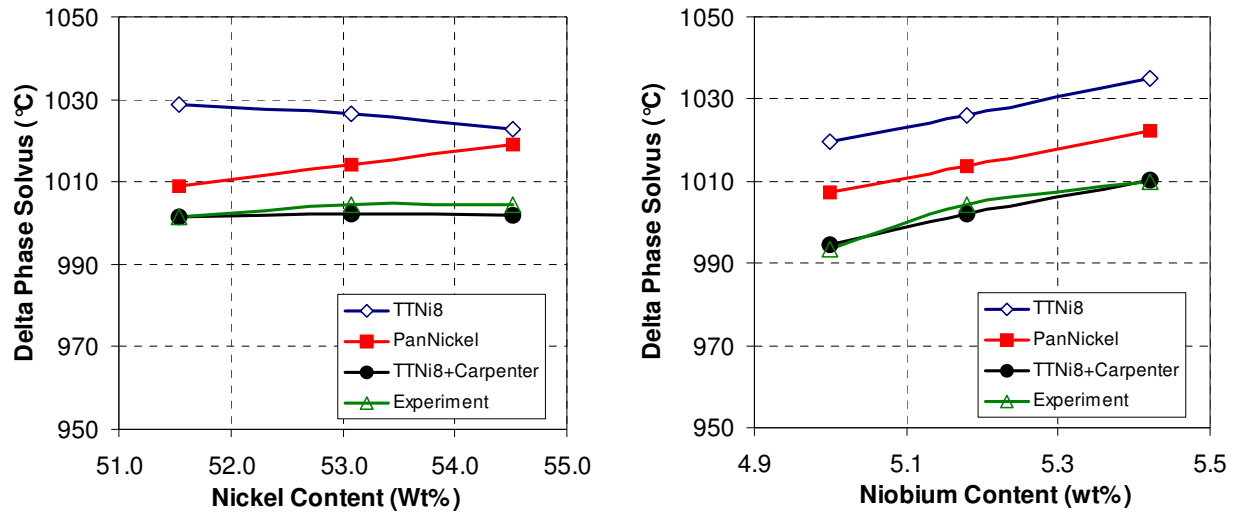


Figure 2. Effect of nickel and niobium contents on delta solvus temperature of alloy 718 – comparison of modeling predictions and experimental results.

PanNickel also predicted that the delta phase solvus temperature increases with nickel concentration, while Ni-DATA (TTNi8) shows the opposite tendency. In terms of the effect of niobium, both databases predict that the delta phase solvus temperature increases with niobium concentration, which agrees with the experimental measurements.

Further studies showed that the disagreement between the predictions and experimental measurements originates from the deficiency of the database in describing the solubilities of niobium in the FCC phase for Ni-Cr-Nb and Ni-Fe-Nb ternary systems. A new database (Carpenter) was developed for the two ternary systems, and much better agreement was achieved by appending the database to the Ni-DATA (TTNi8) database in the thermodynamic calculations (shown as TTNi8+Carpenter in Figure 2).

Mechanical Properties

Figure 3 shows the effect of solution treatment temperature on hardness of alloy 718 with variations in nickel and niobium content. There is no significant effect of nickel content on solution annealing response but, as would be expected, hardness and recrystallization temperature increased with niobium content in the 5.0-5.4% range.

The room-temperature mechanical property data for both treatments are tabulated in Table IV and compositional effects are illustrated in Figures 4 and 5.

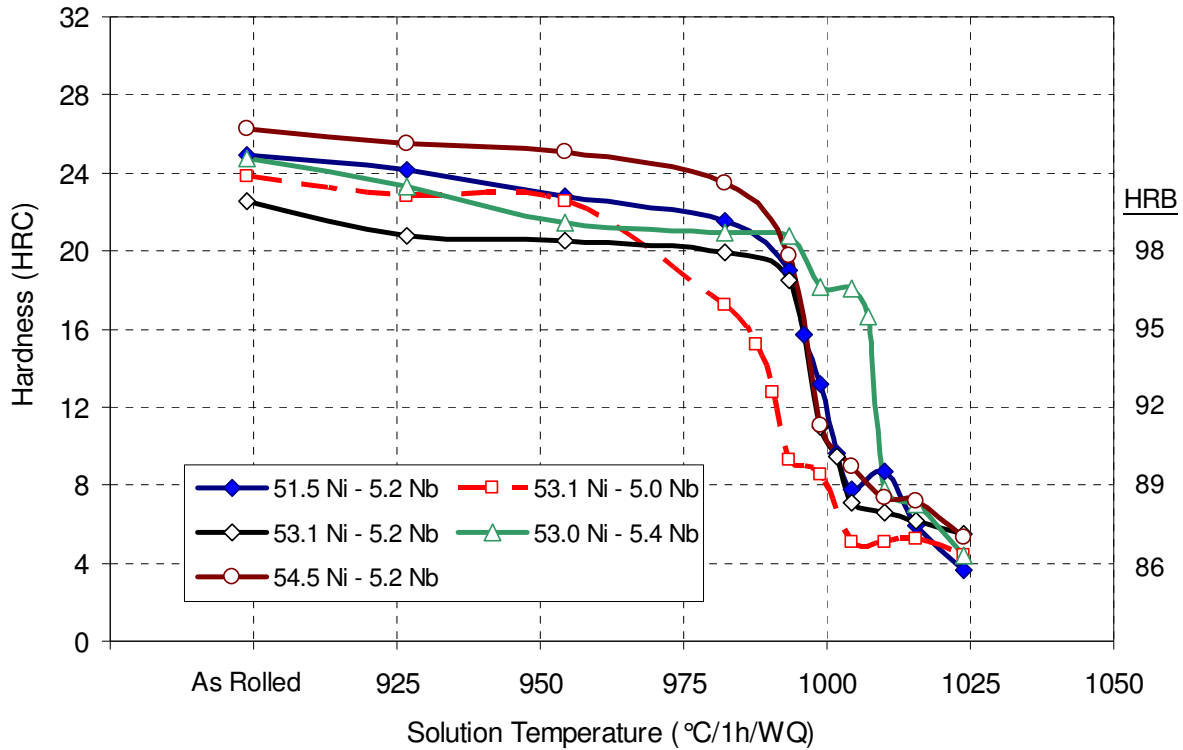


Figure 3. Effect of solution treatment temperature on hardness of alloy 718 with variations in nickel and niobium contents

Table IV. Room-Temperature Mechanical Properties of Alloy 718 With Variations in Nickel and Niobium Contents

		Aerospace Heat Treatment						Oil-Field Heat Treatment					
		954°C/1h/AC + 718°C/8h/FC to 621°C/8h/AC						1024°C/1h/OQ + 785°C/6h/AC					
		Room-Temperature Tensile				Hardness	CVN Impact	Room-Temperature Tensile				Hardness	CVN Impact
		0.2% YS	UTS	Elong.	RA			0.2% YS	UTS	Elong.	RA		
Ni (wt%)	Nb (wt%)	(MPa)	(MPa)	(%-4D)	(%)	(HRC)	(J)	(MPa)	(MPa)	(%-4D)	(%)	(HRC)	(J)
51.5	5.2	1249	1486	18	32	46	22	901	1264	27	46	38	100
"	"	1250	1486	19	34		23	875	1255	28	48		103
53.1	5.0	1229	1471	19	37	45	33	810	1219	30	51	38	111
"	"	1222	1471	19	36		34	816	1216	30	50		114
53.1	5.2	1224	1464	18	32	45	30	894	1251	27	47	39	103
"	"	1230	1465	18	34		28	880	1242	29	53		104
53.0	5.4	1238	1484	19	32	46	24	892	1261	29	51	39	101
"	"	1231	1483	19	39		23	899	1262	29	49		102
54.5	5.2	1228	1490	18	31	46	25	870	1256	30	52	38	107
"	"	1225	1489	18	29		26	870	1253	29	50		108

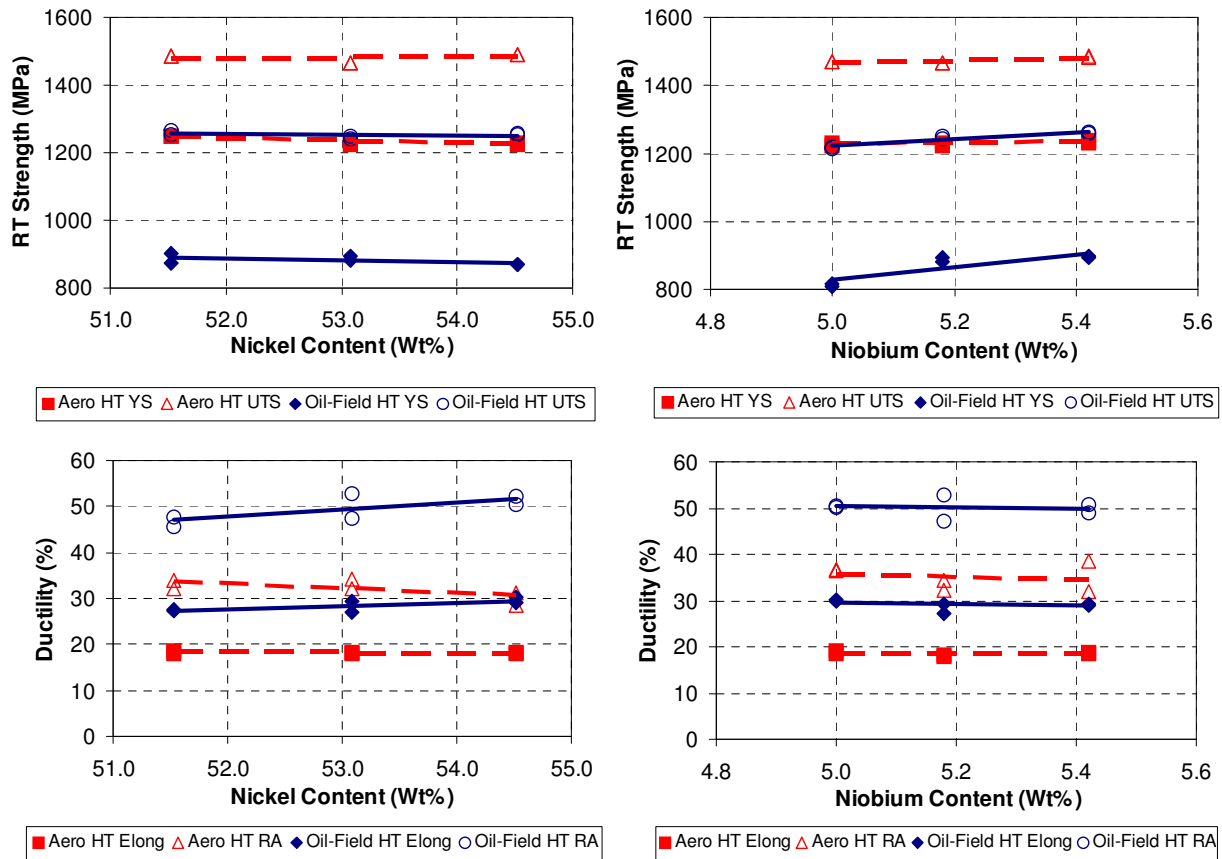


Figure 4. Effect of nickel and niobium contents on room-temp. tensile properties of alloy 718

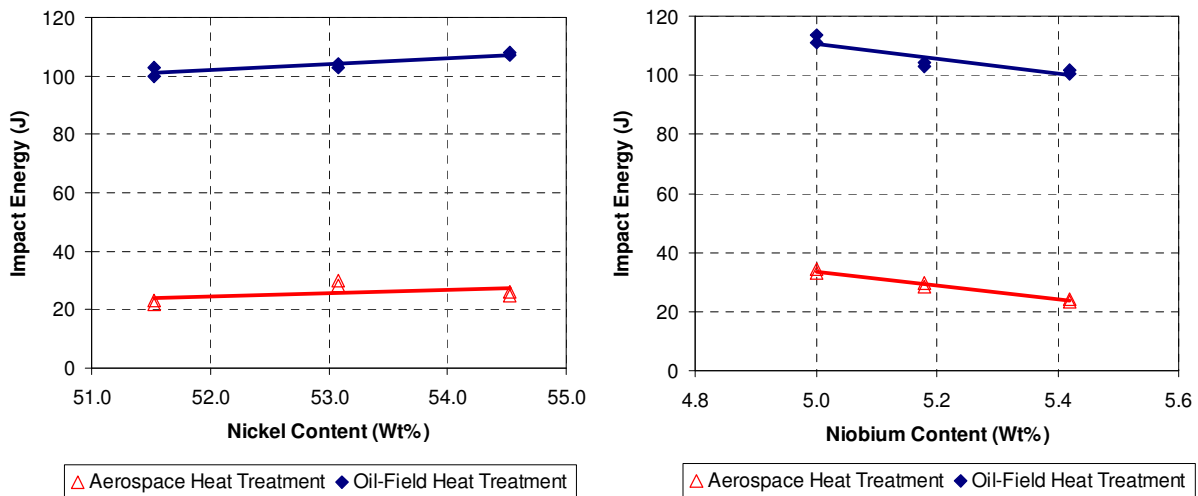


Figure 5. Effect of nickel and niobium contents on Charpy v-notch impact toughness of alloy 718

For both heat treatments, nickel content within the range of 51.5-54.5% had little or no effect on room-temperature tensile properties and possibly a slightly beneficial effect on impact toughness. Niobium within the range of 5.0-5.4% increased strength and reduced toughness. Note that the oil-field heat treatment produced toughness values 3-5 times higher than the aerospace heat treatment. The higher toughness is attributed to lower strength/higher ductility but also to the absence of delta phase in grain boundaries. Delta phase is essential for creep-rupture notch ductility of alloy 718 at high temperatures but can reduce impact and fracture toughness at lower temperatures.

Stress-rupture test results are tabulated in Table V and compositional effects are illustrated in Figure 6. Neither element had significant effects on stress-rupture properties within the ranges evaluated.

Table V. Room-Temperature Mechanical Properties of Alloy 718 With Variations in Nickel and Niobium Contents

		Aerospace Heat Treatment		
		954°C/1h/AC + 718°C/8h/FC to 621°C/8h/AC		
		Stress-Rupture (649°C/758 MPa)		
Nickel (wt%)	Niobium (wt%)	Life (hours)	Elong. (%-4D)	RA (%)
51.5	5.2	59	27	63
"	"	55	28	62
53.1	5.0	68	27	63
"	"	80	27	64
53.1	5.2	80	25	61
"	"	83	28	62
53.0	5.4	75	27	63
"	"	62	26	62
54.5	5.2	45	26	62
"	"	45	28	60

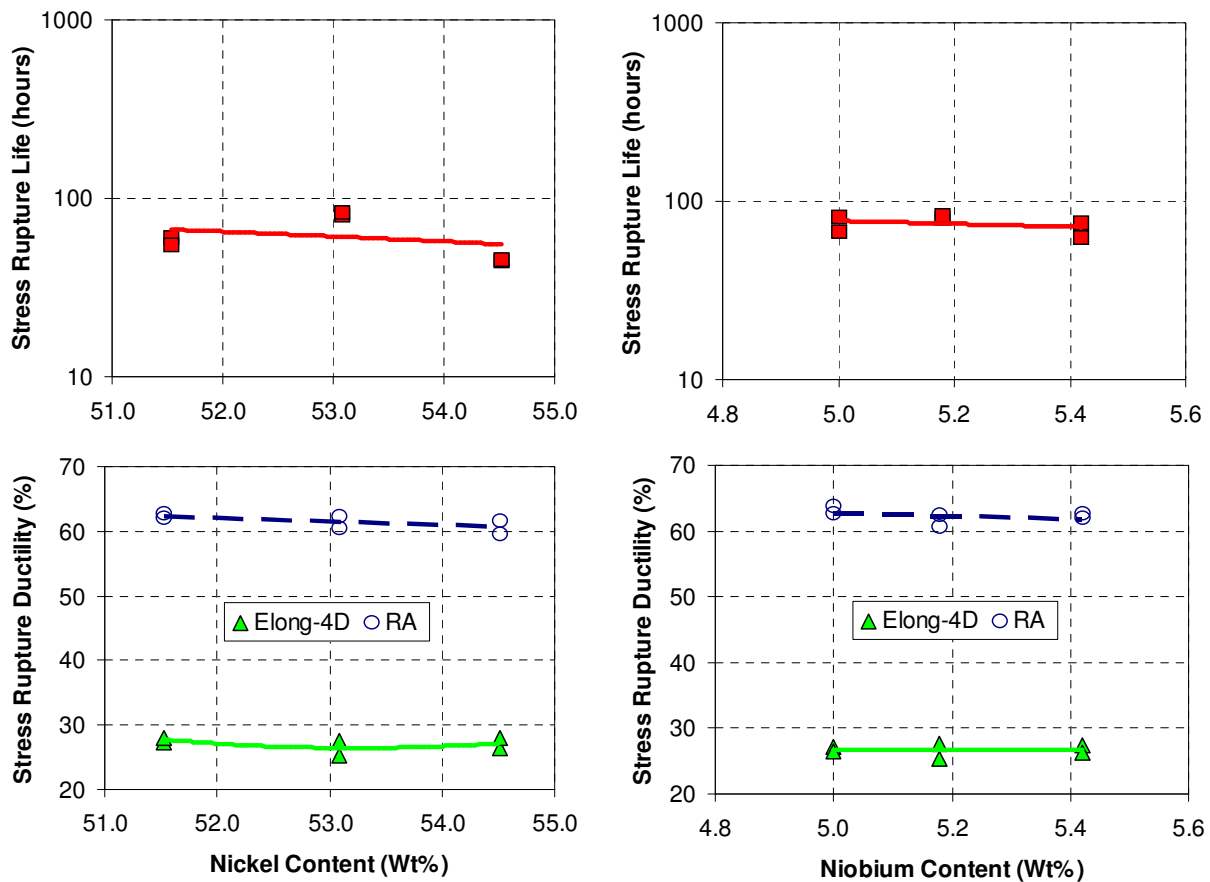


Figure 6. Effect of nickel and niobium contents on stress-rupture properties of alloy 718

Conclusions

This research, supported by prior investigations, shows that nickel content in alloy 718 does not influence the solvus temperature of the delta and gamma double prime phases. Nickel content within the range of 51.5-54.5% also had no significant effect on mechanical properties after heat treatment to aerospace or oil-field specifications. Secondary Laves phase was observed in lower nickel (51.5%) material exposed at 925°C/1700°F (not present with 53.1% Ni), which confirms that there is a minimum critical nickel content, when replaced with iron, to ensure a Laves-free microstructure even in wrought product that was homogenized at the ingot stage. Higher niobium contents did produce the expected effects of higher solvus temperature, higher hardness/strength, and lower ductility/toughness. The oil-field super-solvus solution treatment plus overage heat treatment produces lower strength but higher ductility and toughness compared to the aerospace sub-solvus plus double-age treatment. A newly-developed thermodynamic database resulted in an improvement in the prediction of delta solvus temperature by appending to the Ni-DATA database in the thermodynamic calculations.

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