Elevated Temperature Tensile and Creep Rupture Properties of INCONEL alloy 725

Edward L.Hibner, Howard W. Sizek and Sarwan K. Mannan
Inco Alloys International, Inc.
3200 Riverside Dr.
Huntington, WV 25705-1771

Abstract

INCONEL® alloy 725 (UNS N07725) is a highly corrosion resistant nickel based alloy capable of being age-hardened to high strength levels. In order to determine the potential applicability of alloy 725 in aircraft gas turbine engines, a research program was undertaken to study the solution anneal and age-hardening treatments for required elevated temperature properties. This paper presents the microstructure, tensile and creep rupture properties for several heat treatments.

INCONEL® is a registered trademark of the Inco group of companies.

Superalloys 718, 625, 706 and Various Derivatives
Edited by E.A. Loria
The Minerals, Metals & Materials Society, 1997

Introduction

INCONEL®[1] alloy 725 was originally developed for Oil Patch applications such as well head and subsurface completions.[1-9]. Alloy 725 has been used as pressure-containing, flow-wetted components in subsurface safety valves and as valve components and other down hole components in sour service[10] at temperatures exceeding 350°F(175°C). Alloy 725 is resistant to pitting and stress corrosion cracking (SCC) in Deep Sour Gas Well environments containing NaCl, H_2S and S at temperatures up to about 450 to 500°F (232 to 260°C). Alloy 725 also exhibits excellent corrosion fatigue, pitting and crevice corrosion resistance, in other environments such as seawater. In mineral acids, alloy 725 exhibits corrosion resistance equivalent to alloys 625 and C-276.

With nominally five times the titanium content of alloy 625, alloy 725 can be strengthened by γ' ($Ni_3(Al,Ti)$) and γ'' ($Ni_3(Nb,Ti,Al)$) precipitates by aging between 1150°F and 1350°F. Alloy 725 can be age hardened to strength levels comparable to alloys 706 and 718. This combination of high strength, ductility and excellent corrosion resistance may make the alloy attractive for turbine applications. Alloy 725 has forgeability and machinability comparable to alloys 625 and 718. Weldments on commercially produced alloy 725, have excellent mechanical properties and corrosion resistance[9,11].

Previously, high temperature testing was conducted to provide data for ASME Section VIII, Divisions 1 and 2 Code Case (No. 2217) approval for INCONEL alloy 725 for application to 800°F (427°C). Otherwise limited research has been conducted into the high temperature capabilities of the alloy. The current aging treatments for alloy 725 result in the optimum combination of strength, toughness and corrosion resistance for Oil Patch applications. This research program was conducted to evaluate several solution annealing and age-hardening treatments and the resultant elevated temperature properties.

Procedure

Materials

Material for testing was melted by Vacuum Induction Melting followed by Vacuum Arc Remelting (VIM + VAR) and was taken from two production sources, (1) forged, solution annealed and age-hardened 6 inch (152 mm) diameter bar and (2) hot rolled 3.25 inch (82.5 mm) diameter bar. To study the solution annealing and age-hardening treatments, the 3.25 inch (82.5 mm) material was heat treated in the following conditions prior to testing:

Condition	Heat Treatment
0 & 1	(0)Mill Anneal and Age and (1) Lab Anneal and Age: 1900°F (1038°C)/ 2 hours/ Air Cool + 1350°F (732°C)/ 8 hours/ Furnace Cool 100°F/ hour to 1150°F (621°C) 1150°F (621°C)/ 8 hours/ Air Cool
2	Lab Anneal and Age: 1900°F (1038°C)/ 2 hours/ Air Cool + 1350°F (732°C)/ 4 hours / Air Cool
3	Lab Anneal and Age: 1750°F (954°C)/ 2 hours/ Air Cool + 1350°F (732°C)/ 8 hours/ Furnace Cool 100°F/ hour to 1150°F (621°C) 1150°F (621°C)/ 8 hours/ Air Cool

---

1 INCONEL® is a registered trademark of the Inco group of companies.

Condition	Heat Treatment
4	Lab Anneal and Age: 1750°F (954°C)/ 2 hours/ Air Cool + 1350°F (732°C)/ 4 hours/ Air Cool

The heat treatment, condition numbers 0 and 1, provides the optimum combination of strength, toughness and corrosion resistance for Oil patch applications. Mill anneal implies that the heat treatment was conducted on the full size rod in the manufacturing facility at Inco Alloys International, Inc. (IAII). Lab anneal implies sample blanks cut from the rod were heat treated under laboratory conditions at IAII.

The chemical compositions of the two rods used in this study are as follows:

	Ni	Cr	Mo	Fe	Al	Ti	Nb
6" Rod	Bal	20.81	8.05	7.5	0.17	1.57	3.52
3.25" Rod	Bal	20.75	7.95	8.05	0.16	1.5	3.53

Elevated Temperature Testing

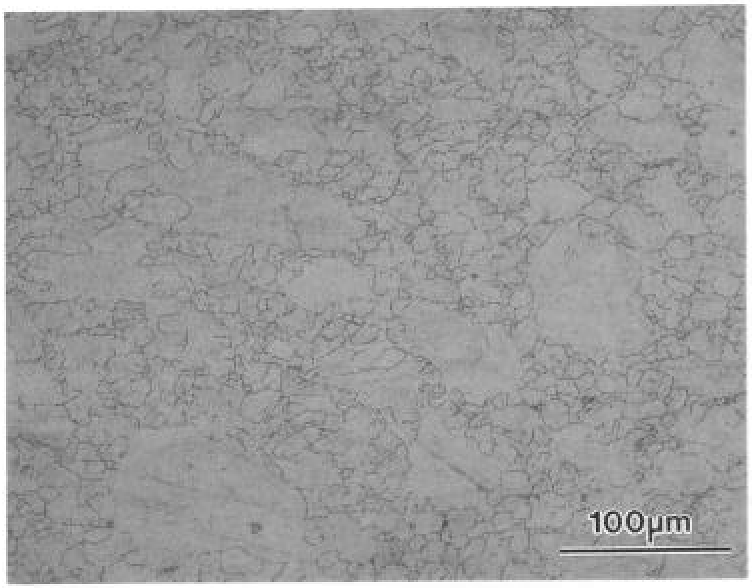
Longitudinal and transverse blanks were cut from the stock and machined into 0.25 inch (6.4 mm) cylindrical samples. Room temperature tensile tests were conducted in accordance with ASTM E 8. Elevated tensile tests were conducted in accordance with ASTM E 21. The stress rupture tests were conducted on low stress ground cylindrical samples with a 0.178 inch (4.5 mm) gage diameter tested at 1000°F (538°C) at an initial stress of 80 ksi (551.6 MPa). The initial stress chosen for testing was too low and as a result incremental loading was necessary to obtain fracture in a reasonable amount of time. After hanging under stress for 3740 hours, the applied stress was incremented by 5 ksi (34.5 MPa) every 48 hours. The temperature control was in accordance with ASTM E 139. Stress rupture testing was conducted in constant load frames.

Results

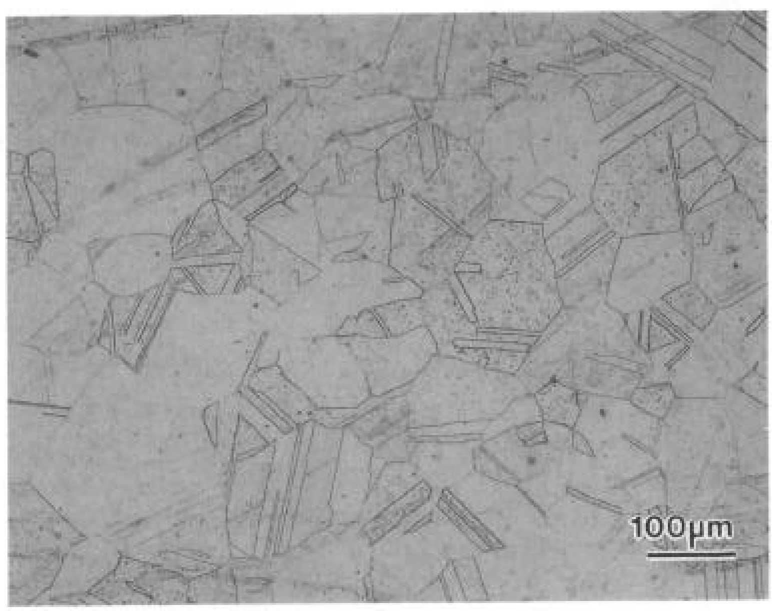
Metallographic Analysis

The material annealed at 1900°F (1038°C) had a normal grain size distribution of ASTM #3. The samples from the hot worked and the hot worked and 1750°F (954°C) annealed material at had a grain sizes ranging from ASTM #5 to ASTM #10 with the average grain size between ASTM #8 and ASTM # 9. Figure 1a and 1b show the optical micrographs from the as rolled material and a sample annealed at 1900°F (1038°C) respectively. The samples were etched with a solution containing three parts hydrochloric acid, one part nitric acid and two parts glycerine. Although the annealed samples were aged at 1325°F (732°C), the grain size did not change during aging.

Figure 2a and 2b are the TEM micrograph and accompanying selected area diffraction pattern of mill annealed and aged alloy 725 (heat treatment 0). Final thinning of foils for transmission electron microscopy was done by jet polishing with 10% perchloric acid in methanol at 32 volts and at -40°F(-40°C). This micrograph along with the accompanying selected area diffraction (SAD) pattern, show the γ'', tetragonal DO_{22} as well as the smaller γ', ($L1_2$) particles. Although not clearly visible in this TEM brightfield micrograph, the additional diffraction spots in the SAD pattern indicate the presence of the γ' precipitates.

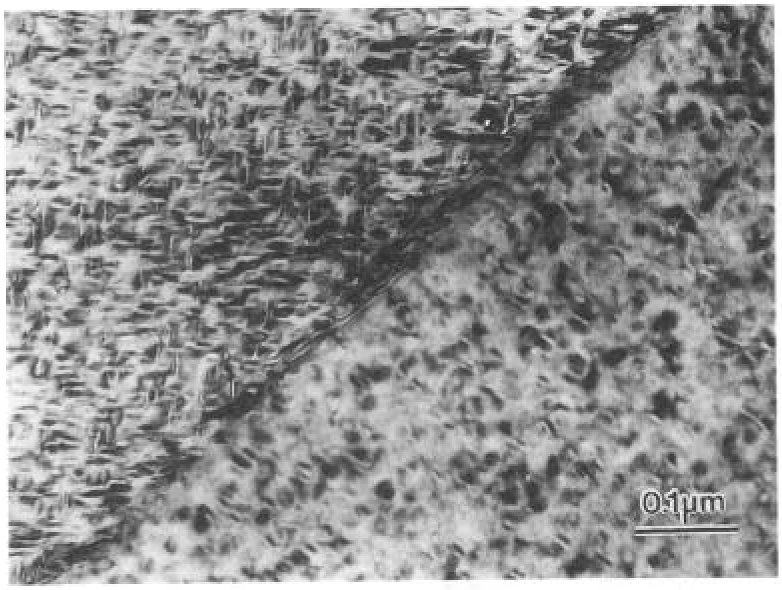


(a)

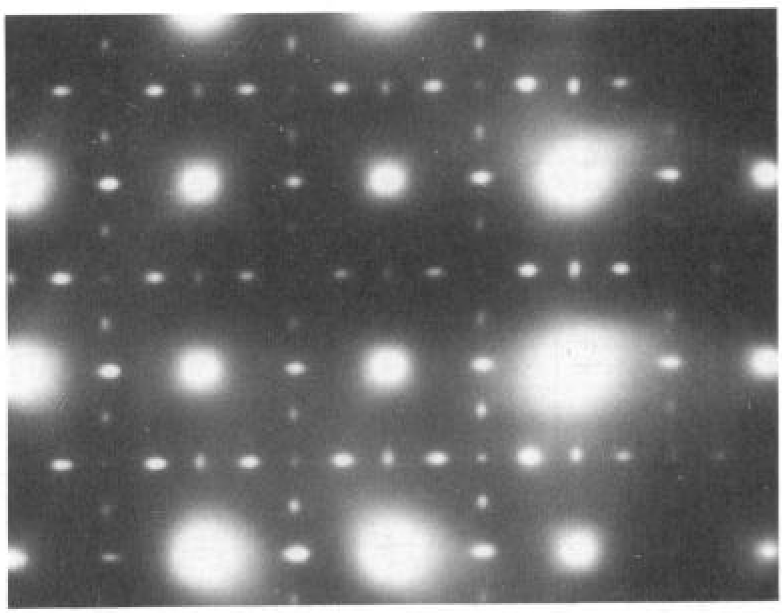


(b)

Figure 1. Optical micrographs of transverse section of 3.25 inch (82.5 mm) alloy 725 rod as: a) hot rolled and b) hot rolled and annealed at 1900°F (1038°C).



(a)



(b)

Figure 2. (a) Transmission electron micrograph (b) and selected area diffraction of alloy 725 annealed at 1900°F(1038°C)/ 2 hours/ and air cooled and then aged at 1350°F(732°C) for 8 hours then furnace cooled at 100°F(38°C)/ hour to 1150°F(621°C) for 8 hours and then air cooled.

Tensile Properties as a Function of Heat Treatment and Test Temperature

Table I displays the longitudinal and transverse tensile properties for the 6 inch (152 mm) diameter forged, mill annealed plus age-hardened bar, i.e., heat treatment 0.

Table IIa and Table IIb display the elevated temperature tensile properties for the 3.25 inch (82.5 mm) diameter hot rolled bar, evaluated in the previously described Heat Treated Conditions 1 to 4.

Table I Elevated Temperature Tensile Properties for INCONEL alloy 725, 6 inch (152 mm) Diameter Forged, Mill Annealed plus Age-Hardened Bar

Test Temperature °F(°C)	Yield Strength ksi (MPa)	Tensile Strength ksi (MPa)	Elongation %	Orientation
72 (22)	123.3 (850)	181.4 (1251)	31.2	Longitudinal
1100 (593)	117.0 (807)	155.1 (1070)	35.8	Longitudinal
	121.1 (835)	153.3 (1057)	31	Transverse
1200 (649)	116.7 (805)	155.6 (1073)	47.6	Longitudinal
	113.5 (783)	152.7 (1053)	38.2	Transverse
1300 (704)	106.1 (732)	137.3 (947)	28.8	Longitudinal
	103.6 (714)	134.0 (924)	22.5	Transverse

Results in this table are the average of two tests.

Table IIa Elevated Temperature Tensile Properties for INCONEL alloy 725, 3.25 inch (82.5 mm) Diameter Hot Rolled Bar Annealed at 1900°F (1038°C)

Heat Treated Condition	Test Temperature °F (°C)	Yield Strength ksi (MPa)	Tensile Strength ksi (MPa)	Elongation %	Tensile Orientation
1	72 (22)	123.7 (853)	177.7 (1225)	31.9	Longitudinal
		122.5 (845)	176.1 (1214)	32.2	Transverse
	1000 (538)	108.0 (745)	142.8 (985)	34.4	Transverse
	1100 (593)	113.0 (779)	143.9 (992)	33.4	Transverse
	1200 (649)	106.6 (735)	147.2 (1015)	31.8	Transverse
2	72 (22)	102.2 (705)	156.5 (1079)	43.8	Longitudinal
		99.6 (687)	152.4 (1051)	45.1	Transverse
	1000 (538)	80.8 (557)	123.7 (853)	56.6	Transverse
	1100 (593)	84.9 (585)	125.8 (868)	44.8	Transverse
	1200 (649)	86.9 (599)	133.8 (923)	37.2	Transverse

Table IIb Elevated Temperature Tensile Properties for INCONEL alloy 725, 3.25 inch (82.5 mm) Diameter Hot Rolled Bar Annealed at 1750°F (954°C)					
Heat Treated Condition	Test Temperature °F (°C)	Yield Strength ksi (MPa)	Tensile Strength ksi (MPa)	Elongation %	Orientation
3	72 (22)	155.5 (1072)	203.6 (1404)	19.1	Longitudinal
		157.3 (1085)	200.1 (1380)	16.6	Transverse
	1000 (538)	144.9 (999)	173.7 (1198)	22	Transverse
	1100 (593)	140.7 (970)	177.5 (1224)	23.1	Transverse
	1200 (649)	131.4 (906)	169.0 (1165)	16.1	Transverse
4	72 (22)	131.1 (904)	184.8 (1274)	27.4	Longitudinal
		128.7 (888)	181.4 (1251)	26.3	Transverse
	1000 (538)	121.3 (836)	160.9 (1110)	28.9	Transverse
	1100 (593)	122.2 (843)	164.2 (1132)	27.8	Transverse
	1200 (649)	130.5 (900)	166.7 (1150)	20.9	Transverse

The elevated temperature tensile data form Tables I and IIa and IIb are presented graphically in Figures 3 to 5.

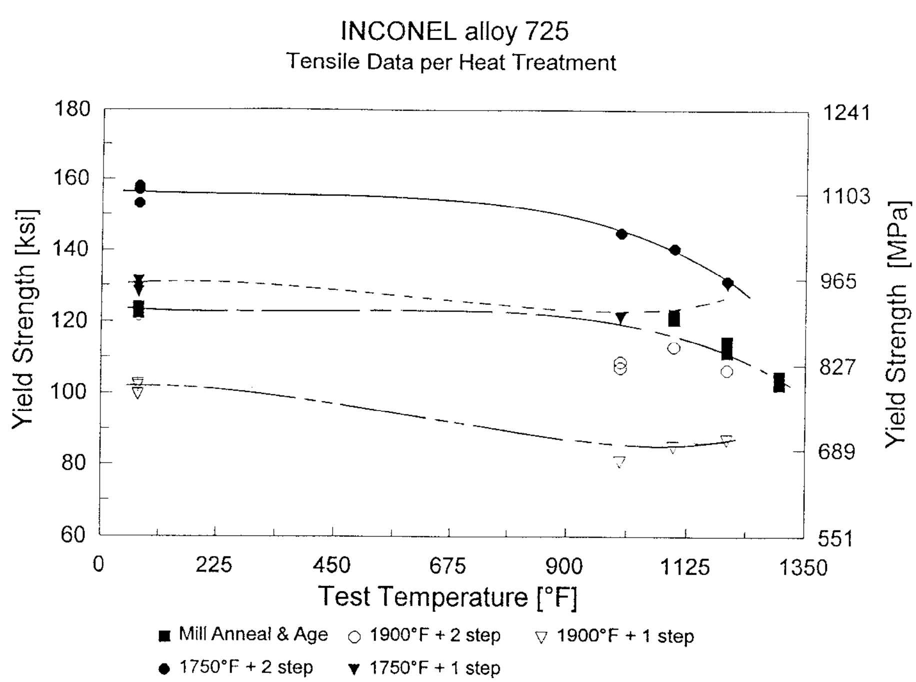


Figure 3 Yield strength versus test temperature from elevated temperature tensile tests of alloy 725.

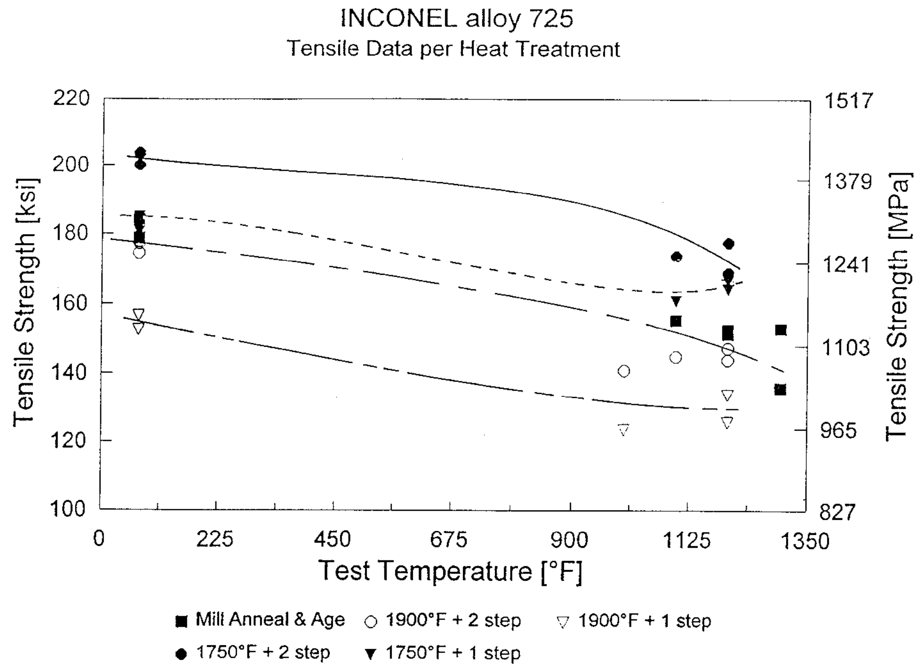


Figure 4 Ultimate tensile strength versus test temperature from elevated temperature tensile tests of alloy 725.

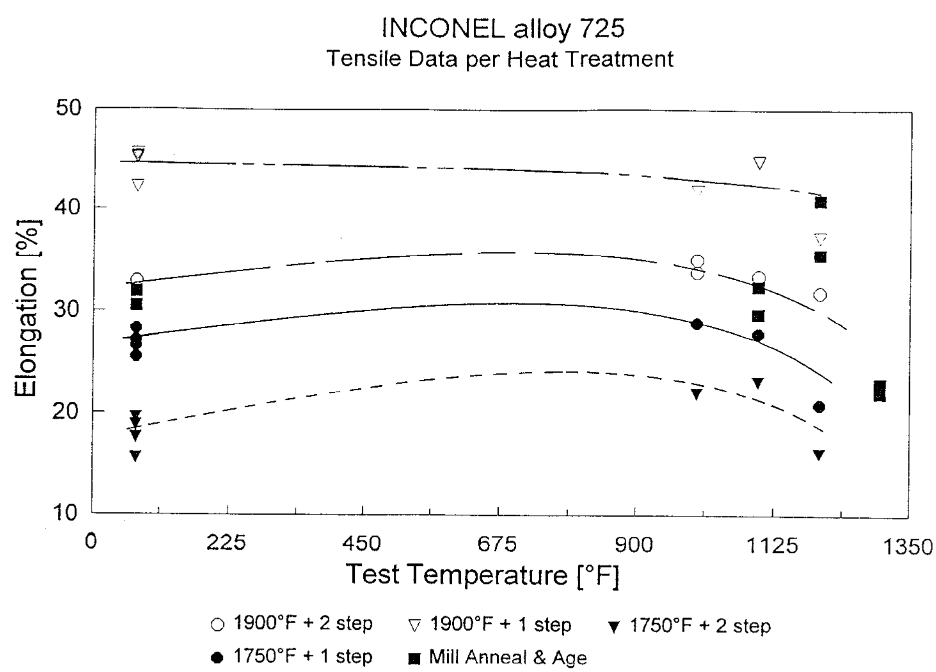


Figure 5 Percent elongation versus test temperature from elevated temperature tensile tests of alloy 725.

Stress Rupture Results

Table III shows stress rupture results for alloy 725, 3.25 inch (8.25 cm) hot rolled bar, evaluated in the previously described Heat Treated Conditions 0 to 4.

Table III Stress Rupture Life for 3.25 inch (82.5 mm) Diameter Hot Rolled Bar, Evaluated in Heat Treated Conditions 0 to 4.				
Heat Treated Condition	Rupture Life hours	Elongation, %	Final Stress, ksi ([MPa])	Time at Final Stress hours
0	4,460	19.4	160 (23.2)	2
	4,510	12.8	155 (1068)	0.1
1	4,419	16.8	150 (1034)	7
2	4,352	29.8	140 (965)	36
3	4,611	8.2	170 (1172)	7
4	4,585	6.2	165 (1138)	29
Initial test conditions: 1000F (538C) at a stress of 80 ksi (551.2 MPa). All stress increases initiated at 3740 hours and increased at 5 ksi (34.5 MPa) every 48 hours.				

Discussion

Elevated Temperature Tensile Properties

The yield strength of alloy 725 hot rolled 3.25 inch (82.5 mm) bar, annealed at 1750°F (954°C) and aged, is between twenty and thirty percent higher than the material annealed at 1900°F (1038°C) and aged at the same times and temperatures. See Figures 3 and 4. The exceptional yield strength observed in the 1750°F (954°C) annealed material, is most likely a combination of the fine grain size and the enhanced aging, resulting from the residual work left in the material after annealing.

Hot rolled alloy 725 bar annealed at 1750°F (954°C) and aged has yield and tensile strengths nearly equal to alloy 718 hot rolled bar, although at these strength levels the ductility for alloy 725 is slightly less than alloy 718. See Tables IIa and IIb. As in any alloy system, a balance exists between strength and ductility. In alloy 725, the strength/ductility balance can be shifted depending on the heat treatment used.

Table IV INCONEL alloy 625 Tensile Properties			
Temperature °F (°C)	Yield Strength ksi (MPa)	Tensile Strength ksi (MPa)	Elongation %
72 (22)	71	140	50
1200 (649)	60	130	38

Alloy 725 annealed at 1900°F(1038°C) has yield strengths thirty to forty percent higher than alloy 625 annealed rod, and tensile strengths twenty to thirty percent higher at ambient and elevated temperatures (Table IV). Both alloy 725 annealed at 1900°F(1038°C) and alloy 625 have comparable elevated temperature tensile elongations. The room temperature ductility of alloy 725 is, however, lower than alloy 625.

The six inch diameter forged bar had tensile properties similar to the 3.25 inch diameter hot rolled rod, indicating that the strength and structure can be maintained in a range of stock sizes.

Stress Rupture

The results of the stress rupture testing of alloy 725, 3.25 inch (82.5 mm) bar are given in Table III. While this is not a constant load test, comparisons of elongations can be made between the differing heat treatments. The stress rupture ductility of the alloy 725 bar annealed at 1900°F (1038°C) is two to three times better than the material annealed at 1750°F (954°C). The elongations from the material annealed at 1900°F (1038°C) are good considering the amount of time under stress at temperature.

Summary

In various heat treated conditions, INCONEL alloy 725 (UNS N07725) exhibited elevated temperature properties significantly higher than alloy 625. This strengthening can be attributed to the Nb and increased Ti contents, which form the γ' and γ'' strengthening precipitates during aging. The increased precipitation kinetics may be attributed to the increased titanium levels. The excellent corrosion resistance of alloy 725 can be attributed to the the Cr and Mo contents.[1-10] Further research will be conducted on the solution anneal and age-hardening treatments to optimize elevated temperature properties of alloy 725.

REFERENCES

1. E.L.Hibner, "A New Age Hardenable Corrosion Resistant Alloy for Deep Sour Gas Well Service," Proceedings of the Ninth International Conference on Offshore Mechanics and Arctic Engineering, ed. M.M.Salama, (New York, NY: ASME), III (B) (1990) 631-638.

2. E.L.Hibner, "A New Age-Hardenable Corrosion Resistant Alloy for Deep Sour Gas Well Service," Paper No. 50, Corrosion '90, Las Vegas, NV, April 1990, (Houston, TX: NACE, 1990).

3. E.L.Hibner, "Corrosion Behavior of a New Age Hardenable Alloy for Oil Field Applications," Proceeding of the First International Symposium on Environmental Effects on Advanced Materials, ed. R.D.Kane, (Houston, TX: NACE, 1991), 14-1 to 12.

4. E.L.Hibner, "A New Age Hardenable Corrosion Resistant Alloy for Deep Sour Gas Well Service," Superalloys 718, 625 and Various Derivatives, ed. E.A.Loria, (Warrendale, PA: TMS, 1991), 895-904.

5. E.L.Hibner, "Corrosion Behavior of Age-Hardenable Alloy UNS N07725 for Oil Field Applications," Paper No. 18, Corrosion '91, Cincinnati, OH, March 1991, (Houston, TX: NACE, 1991).

6. E.L.Hibner and M.N.Maligas, "High Strength Weld Overlay for Oil Patch Applications," Paper No. 144, Corrosion '93, March 1993, New Orleans, LA, (Houston, TX: NACE, 1993).

7. E.L.Hibner and R.H.Moeller, "Corrosion-Resistant Alloys UNS N09925 and N07725 for Oilfield Applications," Paper No. OTC 7206, Offshore Technology Conference, May 1993, (Houston, TX: OTC, 1993).

8. E.L.Hibner and D.B.O'Donnell, "Corrosion Resistant INCONEL alloy 725 Weld Overlay," Symposium on Superalloys 718, 625, 706 and Various Derivatives, ed. E.A.Loria, (Warrendale, PA: TMS, 1994), 893-902.

9. E.L.Hibner and M.N.Maligas, "High Strength and Corrosion Resistant Alloy Weld Overlays for Oil Patch Applications," Paper No. 52, Corrosion '95, Orlando, Fl, March 1995, (Houston, TX: NACE, 1995).

10. NACE Publication 1F192, "Use of Corrosion Resistant Alloys for Resistance to Environmental Cracking in Oilfield Environments," (Houston, TX: NACE, 1992).

11. M.C. Maguire and J.R. Micheal, "Weldability of alloy 718, 625 and Variants," Superalloys 718, 625, 706 and Various Derivatives, ed. E.A.Loria, (Warrendale, PA: TMS, 1994), 881-892.