

LONG TERM THERMAL STABILITY OF INCONEL ALLOYS 718, 706, 909, AND WASPALOY AT 593°C AND 704°C

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Abstract

This paper presents a study of the isothermal stability of INCONEL* alloys 718, 706, 909, and WASPALOY**. Standard annealed and aged materials were exposed at 593°C up to 10,000h and at 704°C up to 5,000h. The exposed materials were tested for room temperature tensile, room temperature impact, and high temperature tensile. The strength of alloy 909 degraded on 593°C exposure but 706 and 718 retained their strength. At 704°C exposure, WASPALOY had the best stability in terms of high

temperature strength whilst 909 was the poorest and retained only 50% of its strength. On 704°C exposure, alloy 706 and WASPALOY retained their room temperature impact strength better than alloy 718. The tested materials were subjected to optical microscopy to develop an understanding of the reasons for the above results.

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** WASPALOY is a trademark of the United Technology Corporation

Introduction

Materials used in gas turbines must have good high temperature microstructural stability. To utilize their maximum potential, precipitation strengthened superalloys are used in the turbine at intermediate temperatures where phase transformations and growth kinetics are rather sluggish. Since the evaluation of intermediate temperature stability requires long term exposure and extensive testing, this data is rare. Although thermal stability of alloy 718 is extensively characterized [1-4], the literature lacks comparative stability data on the Ni-base superalloys.

Dispersion strengthened materials are prone to the coarsening of their precipitates on intermediate exposure, which degrades their strength. The coarsening rate of γ' precipitates in Ni-base alloys was found to relate to coherency strain [5]. Increasing the Ti/Al ratio was found to increase the coherency strain and the coarsening rate in a Ni-based alloy [6]. In a multicomponent system like alloy 718, almost all the alloying elements were analytically

detected in γ'/γ'' precipitates [7]. Since the effect of various elements on coherency strain at different atomic levels is not known, it is difficult to accurately predict thermal stability γ'/γ'' in a multicomponent system.

Experimental Procedure

Commercially produced 101.5 mm hot finished round bar of alloy 718 was used as a starting material. For alloys 909, 706 and WASPALOY, commercially produced forging stocks were hot rolled to 12.5 mm flats in the laboratory. Hot worked materials were subjected to the standard annealing and aging procedures as shown in Table I. The AC and FC in Table I stands for air cool and furnace cool. 706-3 and 706-2 denote 3-step and 2-step aged alloy 706 respectively. The final grain size of alloy 909, 718, 706, and WASPALOY were ASTM # 9.5, 7.5, 5, and 8 respectively. The chemical compositions of these alloys are given in Table II.

Table I. Annealing and aging conditions for the tested alloys

Alloy	Annealing	Aging
718	982°C/1h/AC	718°C/8h, FC 55°C/h to 621°C, hold 621°C/8h, AC
706-2	982°C/1h/AC	Same as alloy 718
706-3	982°C/1h/AC	843°C/3h, AC + same age as alloy 718
909	982°C/1h/AC	Same age as alloy 718
WASP	1020°C/4h, Oil Quench	850°C/4h, AC + 760°C/16h, AC

Table II. Chemical Compositions of the tested alloys in weight percent.

Alloy	Ni	Fe	Cr	Co	Mo	Cb	Al	Ti	C	Si
718	53.4	18.0	18.3	0.13	2.97	5.45	0.59	0.92	0.037	0.10
706	41.5	37.3	16.2	0.03	0.05	3.00	0.24	1.52	0.021	0.05
909	38.3	41.4	0.1	12.9	0.03	5.10	0.05	1.56	0.005	0.42
WASP	57.8	0.40	19.6	13.8	4.25	0.00	1.44	2.92	0.018	0.04

Results and Discussions

Rough-machined, annealed plus aged specimens were exposed at 593°C up to 10,000h and at 704°C up to 5,000h. This was followed by room temperature tensile (RTT) and high temperature tensile (HTT) testing. Specimens exposed at 593°C were HTT tested at 649°C and the specimens exposed at 704°C were HTT tested at 704°C. The specimens exposed at 704°C were also subjected to room temperature impact testing.

The tested specimens were examined by optical microscopy to characterize the microstructure. Alloys 718, 909, and WASPALOY were immersion-etched using Kalling's reagent (100ml methanol, 5gm cupric chloride, and 100ml hydrochloric acid). Alloy 706 was swab-etched using Seven-acids etchant (30ml hydrofluoric acid, 60ml acetic acid, 60ml phosphoric acid, 60ml nitric acid, 300ml hydrochloric acid, 30ml sulphuric acid, 30gm anhydrous ferric chloride, and 300ml water).

593°C Exposure:

Figure 1 shows room temperature and 649°C yield strength of alloys 718, 706-3, 706-2, and 909 following 593°C exposure. Room temperature yield strength of alloy 718 increases up to 8,000h exposure but falls back to almost its as-produced strength on further exposure, Table III and Figure 1. Although the yield strength after 10,000h exposure is higher than the as produced yield strength, it is lower than 8000h exposed yield strength by 55MPa. This is in contrast to a reported study where room temperature yield strength continued to be higher for 50,000h exposure at 593°C [4].

Alloy 909 begins to lose its strength after 2000h at 593°C. On 10,000h exposure at 593°C, the loss in strength of alloy 909 is more than 200 MPa, Table I. Although the as produced 649°C yield strength of 706-2 is 70 MPa lower than 706-3, the 649°C yield strength of 593°C/10,000h exposed 706-2 is 100 MPa higher than the exposed 706-3, Table III. This is related to the additional 843°C/3h heat treatment that alloy 706-3 undergoes following annealing, Table I. Room temperature and high temperature elongation of any of the alloys is not significantly affected by 593°C exposure, Table III.

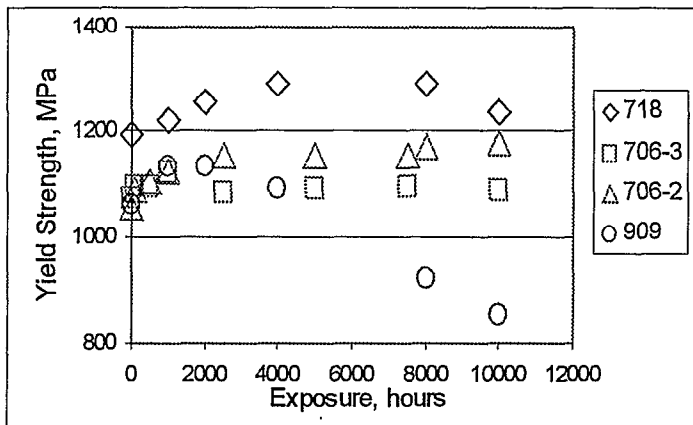


Figure 1a: Room temperature yield strength of materials exposed at 593°C.

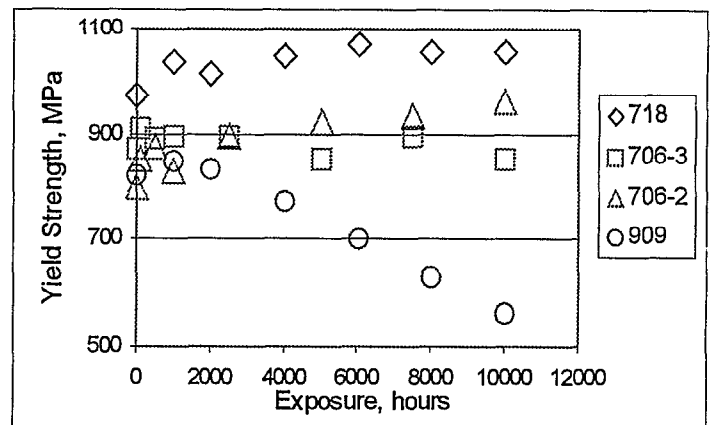


Figure 1b: 649°C yield strength of materials exposed at 593°C.

Table III: Room temperature and 649°C tensile properties following 593°C exposure. Yield strength, tensile strength, percentage elongation, and percentage reduction of area are denoted by YS, UTS, %El, and %RA respectively. The data are the average of two tests. The average values are rounded-off to the whole number.

Alloy	Exposure Condition	Room temperature tensile				649°C tensile			
		YS, (MPa)	UTS (MPa)	%El	%RA	YS, (MPa)	UTS (MPa)	%El	%RA
718	As-produced	1193	1455	16	36	972	1179	31	60
	1000h	1220	1489	14	35	1034	1200	23	62
	2000h	1255	1482	15	35	1014	1200	24	61
	4000h	1289	1496	15	32	1048	1220	22	58
	6000h					1069	1241	21	59
	8,000h	1289	1503	15	34	1055	1241	23	61
	10,000h	1234	1503	14	34	1055	1234	23	60
706 3-Step	As-produced	1077	1319	18	32	874	1025	25	54
	100h	1100	1345	18	36	909	1046	23	54
	500h	1097	1346	17	35	890	1023	25	55
	1000h	1124	1345	18	33	895	1026	23	55
	2500	1085	1346	17	33	896	1026	24	54
	5,000h	1095	1356	16	26	854	985	27	56
	7,500h	1098	1354	17	33	894	1008	24	51
10,000h	1091	1349	18	32	854	997	25	51	
706 2-Step	As-produced	1056	1289	25	53	799	981	31	54
	100h	1091	1313	25	53	852	1030	29	60
	500h	1106	1310	24	55	877	1040	29	61
	1000h	1123	1305	24	53	831	1022	29	61
	2,500h	1153	1337	23	51	894	1046	26	58
	5,000h	1154	1342	22	51	920	1054	27	58
	7,500h	1172	1356	22	51	932	1066	25	57
10,000h	1178	1357	23	50	960	1064	24	57	
909	As-produced	1062	1351	12	33	821	972	26	54
	1,000h	1138	1414	11	34	848	972	19	45
	2,000h	1138	1386	11	35	834	986	19	66
	4,000h	1096	1365	11	31	772	931	23	62
	6,000h					703	890	23	61
	8,000h	924	1262	11	28	628	821	27	69
	10,000h	855	1214	10	22	559	766	29	71

704°C Exposure: Figure 2 shows room temperature and 704°C yield strength of 718, 706-3, 706-2, 909, and WASPALOY exposed at 704°C for up to 5,000h. All the alloys lose their strength after 704°C exposure. Alloy 909 has the poorest thermal stability of all these alloys. After 500h exposure, the loss of strength for alloy 909 was more than 300 MPa, Figure 2. WASPALOY had the best stability in terms of retention of strength. Loss of yield strength at 704°C for WASPALOY and 718 following 704°C/5,000h exposure was 100 MPa and 350 MPa respectively, Table IV. Yield strengths at 704°C of the 704°C/5,000h exposed 706-3 and 706-2 were comparable. Elongation of all the alloys at 704°C increases with the initial 1000h exposure and then saturates on further exposure, Figure 3a. Room temperature elongation is essentially unaffected by exposure at 704°C, Figure 3b.

The decrease in strength of alloys 718, 706, 909, and WASPALOY after 704°C exposure is presumably related to the coarsening of γ'/γ'' precipitates. Coarsening of γ'/γ'' precipitates is a diffusion controlled process. The coarsening rate depends on matrix/particle lattice parameter mismatch, which is responsible for γ/γ' and γ/γ'' interface energy [8]. The higher the lattice mismatch, the higher the growth rate [8]. In the present study, the better stability of WASPALOY compared to alloy 718 is probably related to its lower lattice mismatch. The lattice mismatch of WASPALOY and alloy 718 are +0.30% and +0.80% respectively [9]. Increasing the Ti/Al ratio was found to increase the lattice mismatch and the coarsening rate in a Ni-base superalloy [6]. The decrease in coarsening rate on increase of Cr from 10 wt% to 37 wt% in a Ni-Cr-Ti-Al alloy was attributed to a high

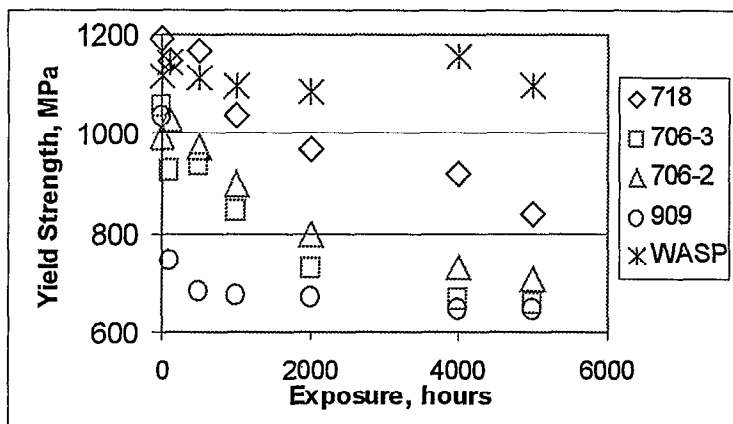


Figure 2a. Room temperature yield strength of the materials exposed at 704°C.

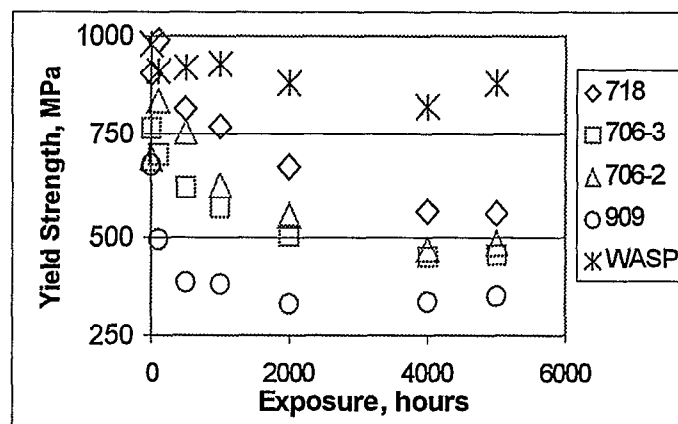


Figure 2b. 704°C yield strength of the materials exposed at 704°C

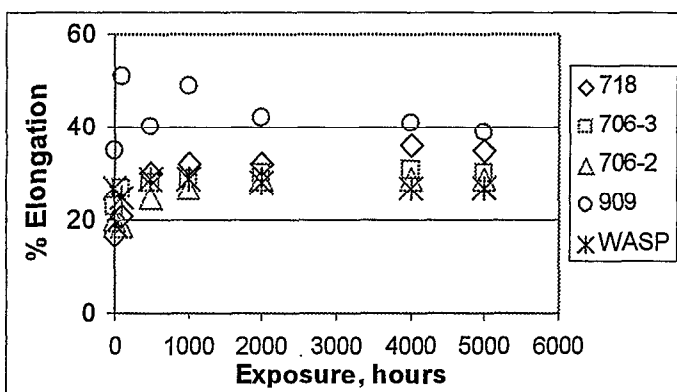


Figure 3a: 704°C elongation of materials exposed at 704°C.

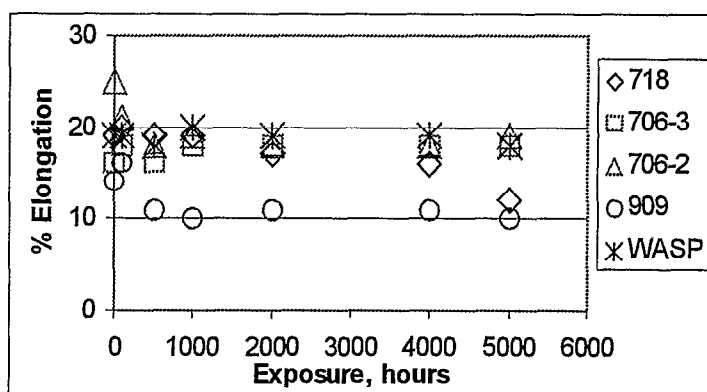


Figure 3b. Room temperature elongation of the material exposed at 704°C.

partitioning coefficient and reduction of coherency strain [5]. A very high partitioning coefficient for an element for γ' reduces the equilibrium concentration in the matrix which decreases the driving force for growth [5]. The addition of 5.5 wt% Mo was found to decrease the coherency strain and retard the coarsening rate of γ' in a Ni-Cr-Ti-Al alloy [10]. The lattice mismatch of alloys 706 and 909 are not known. Thermal stability data of alloy 706 and 909 presented in this investigation will be useful in future to see if the lattice mismatch trend can be extended to these alloys.

Impact Strength Following 704°C Exposure:

Table V shows room temperature impact strength of alloys 718, 706, 909, and WASPALOY following 704°C

exposure. The impact values of as produced and 704°C/5000h exposed alloy 909 are 15 and 10 joules respectively. The impact strength of 706-3 and WASPALOY are essentially unaffected by the exposure. Alloys 718 and 706-2 lose their impact strength on exposure. The impact strength of as produced 706-2 is 111 joules. The impact value of the 704°C/5000h exposed alloy 706-2 is 39 joules which is quite respectable for a precipitation strengthened material. However, the as produced and 704°C/5000h exposed impact strength of alloy 718 are 50 and 9 joules respectively. The low impact value of exposed alloy 718 of 9 joules could be a concern in certain applications. The impact strength versus exposure time plot is shown in Figure, 4.

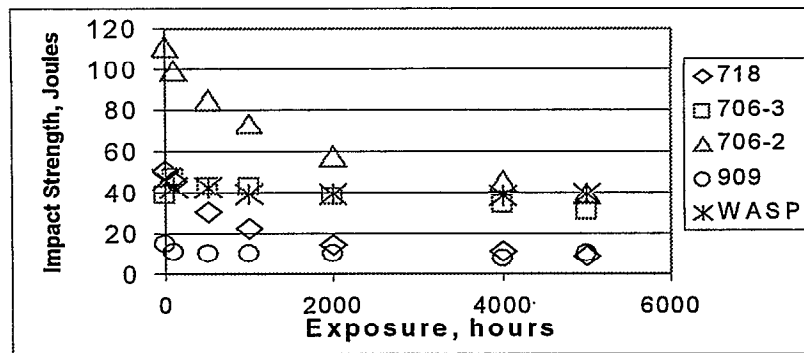


Figure 4. Room temperature impact strength of the materials exposed at 704°C.

Table IV: Room temperature tensile and 704°C tensile properties following 704°C exposure. Yield strength, tensile strength, percentage elongation, and percentage reduction of area are denoted by YS, UTS, %El, and %RA respectively. The data are the average of two tests. The average values are rounded-off to the whole number.

Alloy	Exposure Condition	Room temperature tensile				704°C tensile			
		YS, (MPa)	UTS (MPa)	%El	%RA	YS, (MPa)	UTS (MPa)	%El	%RA
718	As-produced	1192	1352	19	49	904	998	17	29
	100h	1150	1358	20	49	991	1082	21	45
	500h	1167	1377	19	38	816	987	30	68
	1000h	1037	1325	19	44	771	944	32	72
	2000h	969	1289	17	36	671	876	32	66
	4000h	921	1249	16	29	561	811	36	80
	5000h	840	1223	13	17	556	817	35	76
706 3-Step	As-produced	1055	1283	16	30	767	885	23	50
	100h	927	1233	18	39	703	816	27	56
	500h	941	1236	16	38	623	763	28	60
	1000h	847	1177	18	34	572	732	29	58
	2000h	733	1111	18	30	501	685	30	61
	4000h	667	1063	18	29	449	642	31	63
	5000h	662	1056	18	30	457	643	30	60
706 2-Step	As-produced	992	1231	25	56	690	845	20	23
	100h	1029	1264	21	52	834	881	19	47
	500h	974	1236	18	50	759	847	25	62
	1000h	902	1199	19	46	627	767	27	65
	2000h	801	1140	18	35	552	714	29	62
	4000h	752	1097	18	32	468	670	29	59
	5000h	709	1078	19	30	485	679	29	60
909	As-produced	1035	1341	14	39	676	776	35	72
	100h	747	1102	16	29	491	625	51	92
	500h	685	996	11	19	383	556	40	94
	1000h	677	965	10	18	377	530	49	95
	2000h	672	963	11	18	330	510	42	89
	4000h	650	949	11	18	332	476	41	95
	5000h	649	942	10	15	348	495	39	93
WASP	As-produced	1118	1461	19	31	979	1105	27	53
	100h	1145	1489	19	37	910	1129	25	39
	500h	1112	1478	18	29	922	1112	29	55
	1000h	1095	1461	20	38	929	1102	29	57
	2000h	1086	1462	19	37	879	1086	28	50
	4000h	1156	1486	19	49	823	1077	27	56
	5000h	1098	1462	18	36	883	1088	27	52

Table V Room temperature impact strength (joules) of the materials exposed at 704°C

Exposure Time	Materials				
	718	706-3	706-2	909	WASPALLOY
0	50	39	111	15	46
100h	45	48	99	11	42
500h	30	43	85	10	42
1000h	22	43	73	10	39
2000h	14	38	57	10	39
4000h	11	34	45	8	38
5000h	9	31	39	10	39

Optical Metallography: As produced and 704°C/5,000h exposed specimens of alloy 718, 706, 909, and WASPALLOY were examined with optical microscopy to understand the impact properties. Since microstructural development for these alloys is well documented in the literature [1, 2, 4, 12, 13, 14], morphological similarity was used as the only identification for the observed precipitates. The microstructures of as produced and exposed WASPALLOY are comparable, Figure 5a and 5b. This explains its good retention of impact strength on exposure. The grain boundaries of as produced 706-2 are completely devoid of precipitates whereas the grain boundaries of exposed material contains acicular precipitates, presumably Eta phase [12, 13], Figure 5c and 5d. These precipitates would have been responsible for lowering the impact strength from 111 joules to 39 joules,

Table V. Further, intragranular areas of exposed 706-2 have a mottled appearance (Figure 5d) probably due to the coarse γ/γ'' precipitates. As produced 706-3 contains intergranular Eta phase [12, 13], Figure 5e. On exposure, it grows (Figure 5f) resulting in marginal degradation in the impact strength, Table V. Figure 5g and 5h show the microstructure of as produced and 704°C/5,000h exposed alloy 718. Extensive growth of Delta phase colonies [1, 2, 4], relatively higher carbon (0.037wt% as compared to 0.021wt% in alloy 706, Table II), and formation of α -Cr [15] may have been responsible for lower impact strength of exposed alloy 718. Low impact strength of as produced and exposed alloy 909 is due to the presence of globular Laves phase and acicular Epsilon phase [14], Figure 5i and 5j.

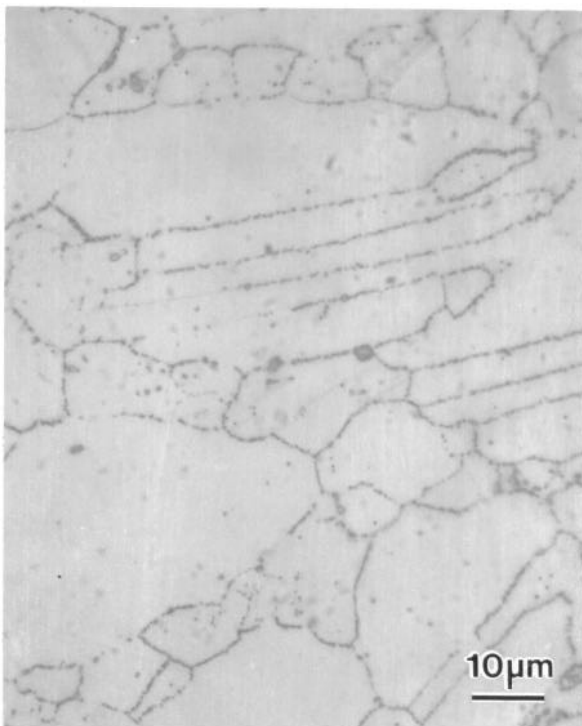


Figure 5a: Optical photomicrograph of as produced WASPALLOY.

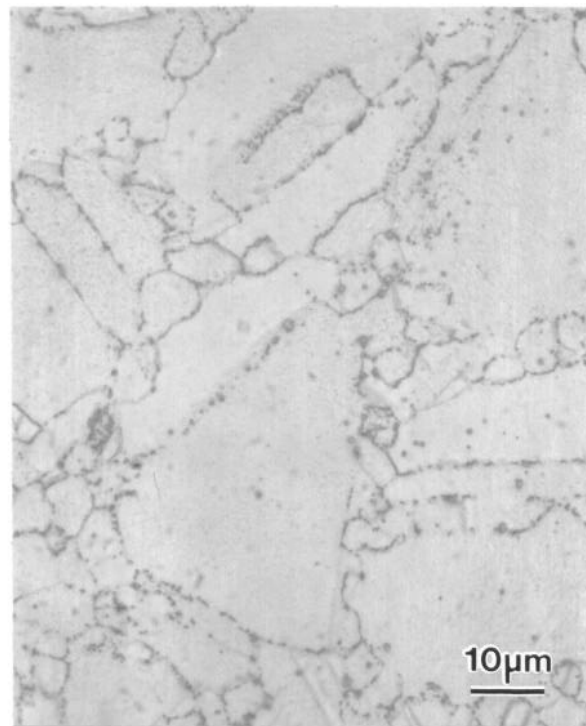


Figure 5b: Optical photomicrograph of WASPALLOY exposed at 704°C for 5,000h.



Figure 5c: Optical photomicrograph of as produced 706-2.

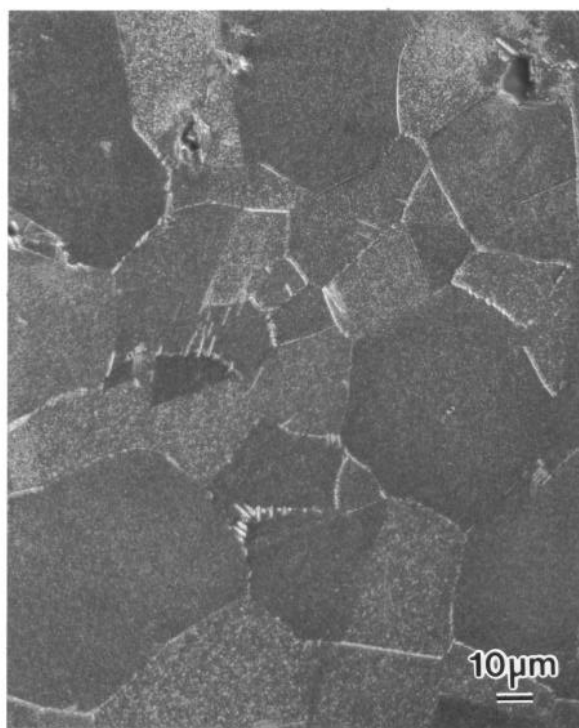


Figure 5d: Optical photomicrograph of alloy 706-2 exposed at 704°C for 5,000h.

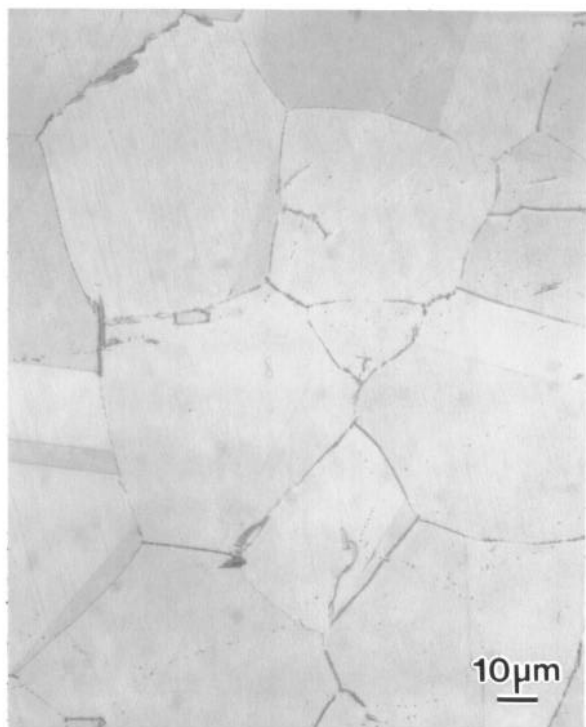


Figure 5e: Optical photomicrograph of as produced alloy 706-3.



Figure 5f: Optical photomicrograph of alloy 706-3 exposed at 704°C for 5,000h.

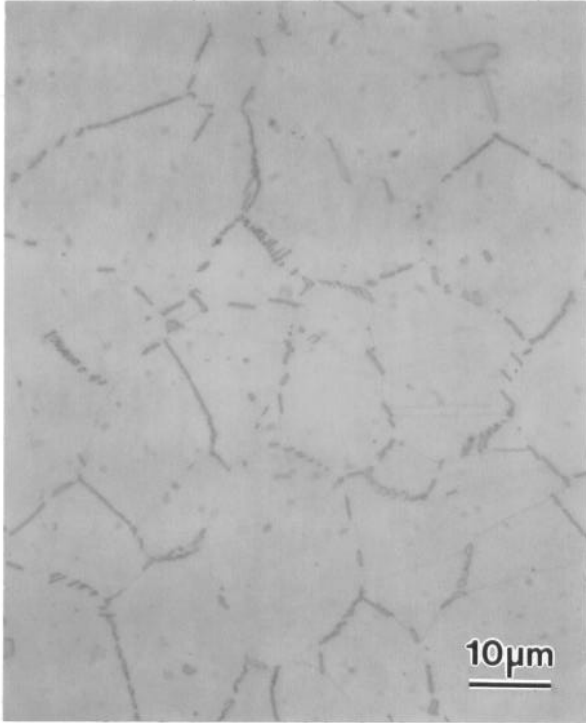


Figure 5g: Optical photomicrograph of as produced alloy 718.

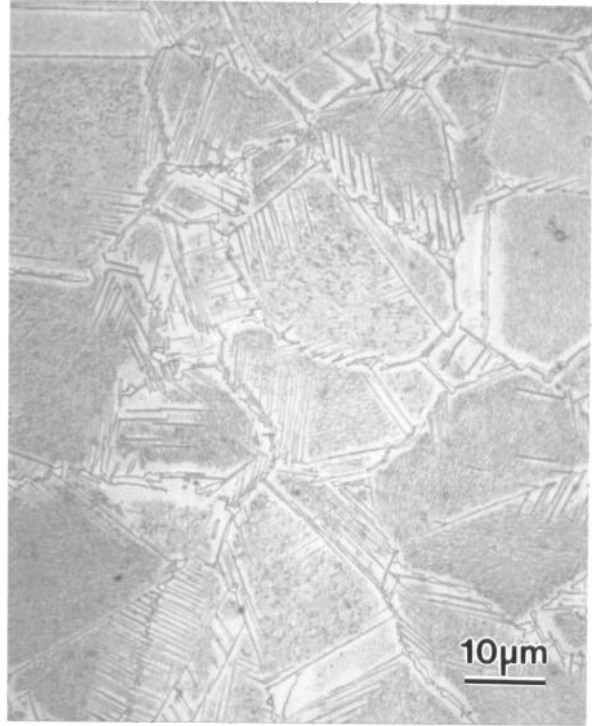


Figure 5h: Optical photomicrograph of alloy 718 exposed at 704°C for 5,000h.

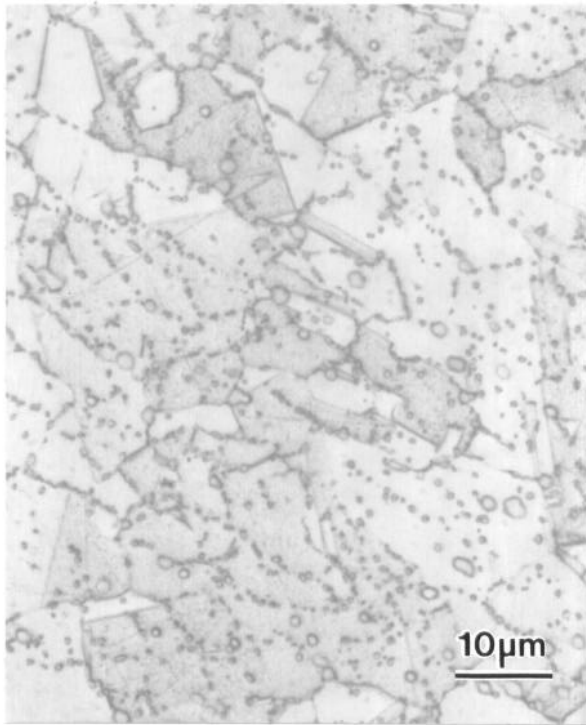


Figure 5i: Optical photomicrograph of as produced alloy 909 showing globular Laves phase.

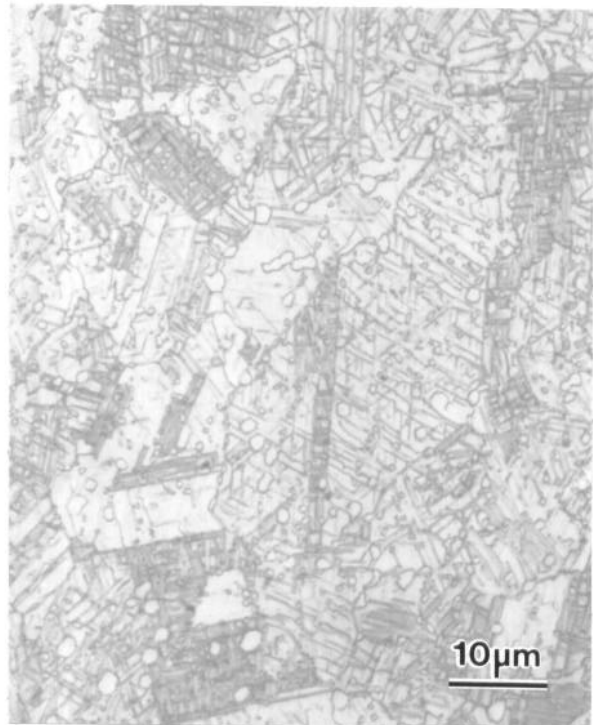


Figure 5j: Optical photomicrograph of alloy 909 exposed at 704°C for 5,000h showing acicular Epsilon phase.

Conclusions

1. Alloy 718 and 706 retain their strength upon 10,000h exposure at 593°C. Alloy 909 begins to lose its strength after 2,000h exposure. Room temperature and 649°C yield strength of 593°C/10,000h exposed alloy 909 is approximately 200 MPa lower than the yield strength of as produced material. Room temperature and 649°C elongations of these materials are not degraded with exposure.
2. Alloy 718, 706, 909, and WASPALOY lose their strength upon 704°C exposure. WASPALOY is the most stable, alloy 909 is the least stable, and alloy 718 and 706 are comparable for their retention of strength on exposure. Room temperature and 704°C elongations of these alloys were not adversely affected after exposure.
3. Room temperature impact strength of WASPALOY and 706-3 (3-step aged) was essentially unaffected upon 704°C/5,000h exposure. Alloy 706-2 (2-step aged) did lose its impact strength but the retained impact strength following 5,000h exposure was respectable (39 joules). Room temperature impact strengths of as produced and 704°C/5,000h exposed alloy 718 were 50 joules and 9 joules respectively. The observed impact strength values were correlated with the microstructure.

References

1. J. W. Brooks and P. J. Bridges, "Metallurgical Stability of INCONEL Alloy 718", Superalloys 1988, Edited by S. Reichman, D. N. Duhl, G. Maurer, S. Antolovich, and C. Lund, TMS, 1988, 33-42
2. J. F. Radavich, "Long Term Stability of a Wrought Alloy 718 Disc", Superalloy 718-Metallurgy and Applications, Edited by E. A. Loria, TMS, 1989, 257-268.
3. S. D. Antolovich, "The effect of Metallurgical Instabilities on the Behavior of IN 718", *ibid*, 647-653.
4. G. E. Korth and C. L. Trybus, "Tensile Properties and Microstructure of Alloy 718", Superalloys 718, 625 and Various Derivatives, Edited by E. A. Loria, TMS, 1991, 437-446.
5. R. F. Decker, "Strengthening Mechanisms in Nickel-Base Superalloys", Paper presented at Steel Strengthening Mechanisms Symposium, Zurich, Switzerland, May 5-6, 1969, 1-24.
6. E. A. Fell, "The Effect of Thermal Treatment on the Constitution of 80-20 Nickel-Chromium Alloys Hardened With Titanium and Aluminum, Metallurgia, Vol. 63, 1961, 157-166
7. M. G. Burke, M. K. Miller, "Precipitation in 718: A Combined AEM and APFIM Investigation", Superalloys 718, 625 and Various Derivatives, Edited by E. A. Loria, TMS, 1991, 337-350
8. T. Tkach et al, "The role of Alloying Elements on γ' Phase Kinetics in Ni-Base Alloys", *High Temperature Materials and Processes*, Vol. 15, No 3, (1996), 195-200.
9. M. Prager and C. S. Shira, "Welding of Precipitation – Hardening Nickel Alloys", (*Welding Research Council Bulletin*, # 128, February 1968)
10. G. N. Maniar, J. E. Bridge Jr., "Effect of Gamma Prime Mismatch, Volume Fraction, and Gamma Prime Morphology on Elevated Temperature Properties of Ni, 20Cr, 5.5Mo, Ti, Al Alloys", *Metallurgical Transaction*, 2 (1971), 95-102.
11. C. E. Jordan, R. K. Rasefske, and A. Castagna, "Thermal Stability of High Temperature Structural Alloys", Long Term Stability of High Temperature Materials, Edited by G. E. Fuch, K. A. Dannemann, T. C. Deragon, TMS, 1999.
12. K. A. Heck, "The Time-Temperature-Transformation Behavior of Alloy 706", Superalloys 718, 625, 706 and Various Derivatives, Edited by E. A. Loria, TMS, 1994, 437-446.
13. J. H. Moll, G. N. Maniar, and D. R. Muzyka, "The Microstructure of 706, a New Fe-Ni-Base Superalloy", *Metallurgical Transactions*, 2 (1970) 2143-2151.
14. K. A. Heck, et al., "The Physical Metallurgy of a Silicon – Containing Low Expansion Superalloy", *Superalloys 1988*, Edited by S. Reichman, D. N. Duhl, G. Maurer, S. Antolovich, and C. Lund, TMS, 1988, 151-160
15. B.A. Lindsley et al., " α -Cr Formation in Alloy 718 During Long Term Exposure: The Effect of Chemistry and Deformation", *Special Metals Corporation, Report # TR-99-008*, May 25, 1999.