**The Ductility Minimum and Its Reversal
with Aging in Cobalt-Base Superalloys**

J. P. Hammond

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ABSTRACT

Good mechanical properties in the face of long-term aging at high temperatures are required of superalloys for nuclear and solar power-conversion applications. Of special concern are losses in ductility and toughness resulting from microstructural instability. This report compares the tensile ductility responses in two cobalt-base superalloys for the solution annealed and aged conditions and endeavors to rationalize results with contemporary concepts.

The cobalt-base superalloys Haynes alloy No. 25 and Haynes alloy No. 188 show a pronounced ductility minimum at 760°C for the solution annealed condition. However, after prolonged (11,000 h) aging at 816°C, copious precipitates form and completely reverse this behavior. These precipitates drastically reduce tensile ductility up to the temperature at which the ductility begins to dip for the solution annealed condition; then the brittle behavior from aging gives way to greatly enhanced ductility.

This behavior in Haynes alloy No. 25 was examined in detail. Tensile properties in the solution annealed and 816°C-aged conditions are correlated with mode of fracture and the amounts, identity, and morphology of the precipitates. The latter were assessed by optical and scanning electron metallography, microhardness, electron microprobe, and x-ray diffraction. The minimum and its reversal are explained by thermally activated processes that began with the onset of recovery.

INTRODUCTION

Among the various criteria for evaluating high-temperature alloys for nuclear and solar energy-conversion applications, good mechanical properties and their retention with prolonged thermal aging rank high. In connection with a broad study¹ of superalloys for such applications, we are measuring the temperature dependence of the tensile properties of several thermally stable alloys (Hastelloy S, alloy 617 and alloy 618) and of some of the earlier less stable alloys such as Haynes alloys No. 25 and 188. The present report addresses the intermediate-temperature ductility loss that occurs in the latter alloys in the solution annealed condition and the effects on ductility of prolonged aging at high temperatures [11,000 h at 816°C (1500°F)].

For these alloys this aging exposure precipitated substantial amounts of carbide and intermetallic phases. These precipitates, as expected, promoted severe embrittlement at low test temperatures, but at the intermediate temperature at which the ductility minimum ordinarily occurs for solution annealed material [$0.5-0.6T_m$ (1033 K)], ductility was restored. To better understand these phenomena, we studied Haynes alloy No. 25 in some detail.

Significance is attached to ductility-minimum behavior as it influences the hot workability of metals.² Others associate it with strain cracking in welds.^{3,4} The ductility reversal with aging could have importance at slower rates of straining, as in creep.⁵

MATERIALS AND METHODS

The investigation was conducted on three heats of Haynes alloy No. 25 (0.01, 0.11, and 0.36% Si)^{*} and one heat of Haynes alloy No. 188. The vendors' chemical analyses and the heat numbers of the alloys are given in Table 1. The chemical composition of each of the various heats

^{*}Heats with varying silicon were included because silicon may accelerate precipitation with aging. However, silicon proved to have little or no effect for the aging conditions we employed.

Table 1. Vendors' Chemical Analyses of Superalloys

Element	Content, wt %, for Each Alloy (Heat) ^a			
	Haynes 25 (186-6-1816)	Haynes 25 (1860-8-1335)	Haynes 25 (B18509)	Haynes 188 (8188-7-0124)
Ni	9.25	10.19	10.59	21.44
Cr	19.77	19.39	20.18	22.27
Fe	2.02	2.82	1.58	1.33
W	14.42	14.96	14.66	14.49
Mn	1.33	1.28	1.70	0.76
C	0.10	0.11	0.078	0.11
Si	0.01	0.11	0.36	0.08
P	0.020	0.013	0.010	0.012
S	0.007	0.002	0.010	0.001
La				0.04

^aBalance cobalt.

conformed to the corresponding materials specifications (AMS 5537C and AMS 5608). Haynes alloy No. 25 and Haynes alloy No. 188 are cobalt base, with the former containing nominally 20 wt % Cr, the latter 22 wt % Cr, and each 14.5 wt % W as major alloying additions. The hardening that occurs in Haynes alloy No. 25 with precipitation of secondary phases is described by Yukawa and Satu,⁶ whereas Herchenroeder et al.⁷ describe precipitation in Haynes alloy No. 188.

The tensile tests were previously conducted on sheet specimens of the alloys nominally 1.5 mm thick with a 25.4-mm-long by 6.35-mm-wide gage section.¹ The long-term (11,000-h) aging was performed in vacuum on machined specimens that had been given a prior solution anneal. The specimens tested in the solution annealed condition were fabricated directly from as-received stock. Surfaces of the original sheet were kept intact in both cases. The tensile testing was performed in air at a crosshead speed of 21 $\mu\text{m/s}$ (0.05 in./min) in a 44.5-kN (10,000-lb) Instron tensile testing machine. The detailed tensile data were previously documented.¹

We have now studied extensively the deformation and aging responses of specimens of Haynes alloy No. 25 tensile tested both as solution annealed and after 11,000 h aging at 816°C. These studies included examinations by optical and scanning electron metallography, micro-hardness tests, x-ray diffraction, and x-ray fluorescence determination of matrix depletion.

RESULTS

The additional tensile data gathered on Haynes alloy No. 25 are given in Table 2. The yield strength, ultimate tensile strength, and total elongation determined previously¹ as a function of test temperature are shown as composite graphs for two of the Haynes alloy No. 25 heats and for Haynes alloy No. 188 in Figs. 1 and 2, respectively. The solution annealed and 11,000-h-aged conditions are compared. The properties for the aged condition are given at room temperature, 316, 760, and 982°C only, sufficient to reflect the significant changes in the deformation and aging responses.

Table 2. Tensile Properties of Haynes Alloy No. 25 After Aging 11,000 h
at 816°C (1500°F) Compared with Solution Annealed Material

Aging Time ^a	Test Temperature		Stress, MPa (ksi)			Elongation, %			Energy to Fracture	
	(h)	(°C)	(°F)	Yield (0.2%)	Ultimate Tensile	Fracture	Total	Uniform	Stable ^b	(J/m ³) (ft-lb/in. ³)
0	24	75		493 (71.5)	996 (144.4)	987 (143.1)	62.5	61.8	62.5	543.1 (6663)
11,448	24	75		398 (57.8)	927 (134.5)	927 (134.5)	10.2	10.2	10.2	87.4 (1072)
11,448	24	75		380 (55.1)	1022 (148.3)	1022 (148.3)	10.8	10.8	10.8	90.9 (1115)
0	316	600		314 (45.5)	852 (123.6)	812 (117.8)	78.1	74.9	77.4	540.6 (6632)
11,448	316	600		329 (47.7)	823 (119.4)	823 (119.4)	20.2	20.2	20.2	147.3 (1807)
11,448	316	600		327 (47.4)	767 (111.2)	767 (111.2)	13.8	13.8	13.8	100.3 (1231)
0	536	1000		279 (40.4)	755 (109.5)	590 (85.6)	78.8	74.9	77.2	
11,448	536	1000		342 (49.7)	748 (108.6)	737 (106.9)	9.2	8.9	9.0	
0	649	1200		252 (36.6)	705 (102.2)	705 (102.2)	66.2	66.2	66.2	
11,448	649	1200		339 (49.2)	842 (122.4)	838 (121.6)	14.7	14.2	14.3	
0	760	1400		251 (36.4)	432 (62.9)	58 (8.44)	21.2	16.2	16.2	71.7 (879)
11,448	760	1400		262 (38.0)	586 (85.0)	475 (68.9)	57.7	16.7	51.5	302.3 (3709)
11,448	760	1400		261 (37.8)	582 (84.7)	463 (67.2)	59.4	16.6	54.5	307.9 (3777)
0	871	1600		221 (32.0)	324 (46.9)	234 (34.0)	40.7	11.4	21.9	
11,448	871	1600		273 (39.5)	338 (49.0)	149 (21.7)	73.9	7.2	30.1	
0	982	1800		185 (26.3)	186 (26.9)	103 (14.9)	43.9	0.3	13.4	68.0 (834)
11,448	982	1800		152 (22.0)	182 (26.4)	114 (16.6)	48.1	6.0	26.1	
11,448	982	1800		73 (10.5)	92 (13.4)	27 (3.9)	75.0	7.7	32.8	106.2 (1303)
0	1093	2000		94 (13.5)	98 (14.3)	65 (9.5)	21.0	1.7	2.4	
11,448	1093	2000		96 (13.9)	102 (14.7)	49 (7.1)	61.9	3.0	39.7	

^aMaterial with no aging time is 1.5-mm (0.059-in.) sheet in heat 1335, solution annealed by vendor. Material with long-time aging is 1.1-mm (0.043-in.) sheet in heat 1816.

^bStrain at the maximum true stress.

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Fig. 1. Comparison of the Effects of Solution Annealing with Those of Aging at 816°C on Yield Strength, Ultimate Tensile Strength, and Total Elongation of Haynes Alloy No. 25. Data on solution annealed specimens are from Haynes alloy No. 25, brochure F-30,041E, Cabot Corporation, Kokomo, Indiana, 1970.

ORNL-DWG 75-18002

Fig. 2. Comparison of the Effects of Solution Annealing with Those of Aging at 816°C on Yield Strength, Ultimate Tensile Strength, and Total Elongation of Haynes Alloy No. 188. Data on solution annealed specimens are from Haynes alloy No. 188, brochure F-30,361B, Cabot Corporation, Kokomo, Indiana, 1973.

Observe that for both alloys the 11,000-h aging resulted in poor ductility at room temperature and 316°C compared with results for the solution annealed condition, but at 760°C ductility is considerably improved. Significantly, this gain of ductility occurs in the temperature region where ductility for the solution annealed condition achieves its minimum and may appropriately be termed "minimum reversal." At 982°C, a temperature at which ductility for the solution annealed condition is well restored, aged ductility remains high. It is noteworthy that while the alloys in the solution annealed and 11,000-h-aged conditions differ markedly in ductility, little difference in either yield strength or ultimate tensile strength is noted.

Although yield strength and ultimate tensile strength varied little between the two metal conditions, the large difference in total elongation suggests important differences in toughness responses between the two conditions, since this quality is a product of strength and ductility. In Fig. 3 the energy of deformation to fracture, as measured from the area under the tensile curve, is shown for Haynes alloy No. 25 in the solution annealed condition and for each of the alloys examined in the aged condition. Observe for Haynes alloy No. 25 that the improvement in ductility cited at 760°C is reflected in an equally impressive toughness. For each of the cobalt-base alloys, toughness in the aged condition achieves by far its highest value at 760°C (the ductility minimum temperature for the solution annealed material) and greatly exceeds that for the solution annealed condition at this temperature.¹ The large drop-off in aged toughness on going to 982°C results from loss of strength rather than ductility at this temperature. It is appropriate, then, in studying the mechanical properties in Haynes alloy No. 25 to focus on the underlying causes of the ductility minimum that occurs for the solution annealed condition and the reversal exhibited with 11,000-h aging at 816°C (1500°F).

Rhines and Wray⁸ first observed that under proper conditions of temperature and strain rate a dip in ductility at an intermediate temperature range occurs in all ductile metals. They pointed out examples in transitional metals and in low-melting and high-melting metals alike. The common characteristic of the minimum is the rapid drop-off of total

Fig. 3. Deformational Energy at Fracture of Superalloys Under Varying Metal Conditions and Test Temperatures. Bar charts give average of two values at 24, 316, 760, and 982°C (75, 600, 1400, and 1800°F) consecutively.

elongation just above the recovery temperature. They postulated that at low temperatures fracture occurs by transgranular crack propagation with extensive deformation and ductility is high. At intermediate temperatures deformation occurs by grain boundary shear and intergranular voids form at triple junctions and grow unhindered to give intergranular failure. This causes the minimum in ductility. At higher temperatures recrystallization occurs simultaneously with intergranular void formation and continuously breaks up the path of intergranular cracking.⁸ Thus, ductility increases again. In reporting findings of high-strain fracture in alloy 600 resulting from hot torsion testing, Shapiro and Dieter² concurred in the generalized observations advanced by Rhines and Wray. Later, Arkoosh and Foire⁹ examined ductility minimum behavior in Hastelloy alloy X (a nickel-base superalloy) and confirmed the conclusions of Rhines and Wray, with one exception. They contended that restoration of

ductility above the minimum results from matrix depletion and that recrystallization occurs only at much higher temperatures.

Whereas the foregoing concepts have been advanced to explain the ductility-minimum phenomenon so prevalent in metals and alloys, little appears in the literature on the reversal effect. Figure 4 more clearly illustrates this effect in Haynes alloy No. 25. In addition to showing the total elongation curves for the solution annealed and 11,000-h-aged conditions, curves labeled "stable elongation" are included. This property is defined here as the elongation at maximum true stress and marks the onset of unstable strain. Significantly, this graph shows that the improvement in ductility at 760°C resulting from 11,000-h aging is largely due to a gain in stable strain.

Fig. 4. Stable and Total Elongation of Haynes Alloy No. 25 as Influenced by Metal Condition and Test Temperature.

In explaining the underlying principles of this graph it is expedient to consider the region between 649 and 871°C since, as we shall see subsequently, the dividing line between the low- and the high-temperature cracking mechanisms is here. A basic transition is that the failure

mode switches from transgranular (TG) to intergranular (IG) for the solution annealed condition and from IG to TG for the 11,000-h-aged condition with rising temperature in this range. Generally TG failure involved large total elongation, as manifested by an extension of grains in the loading direction. On the other hand, IG failure gave a low total elongation with little change in grain shape. A satisfactory explanation of the minimum and its reversal should account for these relationships.

The microstructures at the fracture faces for the various solution annealed and 11,000-h-aged specimens pulled in this transition region are shown in Fig. 5. The fracture modes and total elongation values are given beneath the micrographs.

Microhardness of grain centers just off the fracture surfaces was measured for tests at room temperature and from 649 to 982°C for both the solution annealed and the 11,000-h-aged conditions. Curves of room-temperature hardness versus tensile testing temperature for Haynes alloy No. 25 in the two conditions are compared in Fig. 6.

A final aid to interpreting the results is the following information excerpted from the work of Yukawa and Satu⁶ on the identification and kinetics of phase precipitation in Haynes alloy No. 25. The aging process proposed at 800°C (1472°F) is:

The phase in parentheses is metastable. Age hardening at 800°C and higher is attributed to precipitation of carbides ($M_{23}C_6$ and M_6C) and Laves- Co_2W . At temperatures below 800°C, 100 h or longer is required to initiate precipitation of the Laves- Co_2W phase, whereas the carbides are formed in much shorter periods. The Laves- Co_2W phase is rather fragile and is thought to promote brittleness in Haynes alloy No. 25,¹⁰ whereas the carbide phases generally are tougher. For our aged Haynes alloy No. 25 specimens, a weighing and an x-ray diffraction analysis of residues extracted by electrolysis indicated a total of 14.7 wt % precipitate had formed; approximately half of this was Laves- Co_2W and the remainder unidentified phases, perhaps $\mu\text{-Co}_7\text{W}_6$ or carbides.

Fig. 5. Microstructures at Fracture of Haynes Alloy No. 25 Tensile Specimens Pulled After Solution Annealing or After Solution Annealing and Aging at 816°C. Etch: 100 ml HCl + 12 drops H₂O₂.

Fig. 6. Comparison of Microhardness at Room Temperature After Pulling Solution Annealed and 11,000-h-Aged Haynes Alloy No. 25 Specimens (0.11% Si) at Various Temperatures.

As in previous findings on ductility minimum behavior,^{2,8,9} we found fracture in the solution treated alloy to occur by the usual trans-granular crack propagation mechanisms at temperatures below the transition (649°C and lower), and elongation, accordingly, was high. This will be noted from the microstructure after testing at 649°C [Fig. 5(a)]. Total elongation was 66%, and grains were extended along the gage section.

At the minimum-ductility temperature (760°C) intergranular voids formed at triple junctions as a result of grain boundary shear and eventually grew unimpeded to give early intergranular fracture [Fig. 5(b)].

Total elongation was a low 21%, and grains along the gage section, accordingly, were relatively unextended. This fracture is one of three basic types illustrated by scanning electron microscopy (SEM). Observe in Fig. 7(a) that failure indeed is intergranular, but the distinctive cusps seen at higher magnification indicate high localized plasticity. Similar features prompted Rhines⁸ to coin the expression "plastic brittleness" for this behavior.

The minimum behavior here proved so pronounced that we suspected that strain-induced submicroscopic precipitation hardening (by carbides) and concomitant softening near boundaries by precipitate denudation abetted the cracking. However, the low hardness exhibited in Fig. 6 by the specimen tested at the ductility minimum temperature suggests that this may not be the case. Instead, the intergranular cracking would appear to be aided by the dynamic dislocation recovery mechanism postulated by Sikka.⁴ He suggest that work-hardened grains are preferentially recovered adjacent to grain boundaries as a result of a low vacancy diffusion rate and, as a consequence, the grain boundary sliding that leads to intergranular cracking is facilitated.

At 871°C fracture occurred as a result of predominantly transgranular deformation acting in concert with intergranular cavitation cracking [Fig. 5(c)]. Although the mode of fracture was still intergranular, total elongation was a moderately good 41%, and grains correspondingly were extended along the gage section. At this temperature the vacancy diffusion rate is high, and, accordingly, dislocation entanglements throughout grains are expected to recover as dislocations climb and glide to either annihilate each other or form subgrains. Also, intergranular cracking is delayed by thermal recovery of crack fronts. Unlike the findings of Rhines,⁸ evidence is lacking for recrystallization isolating intergranular cracking and in this way enhancing ductility.

At 982°C the failure mode again was distinctly intergranular, but transgranular deformation obviously contributed substantially to a high 44% total elongation. Here the cracking occurred at triple junctions adjoining the rather bulky constituent that had formed continuously in the grain boundaries.

Y154870

760°C

IG but Ductile (21% T. Elong.)

RT

IG and Brittle (10%)

PULLED AT: 760°C

TG and Ductile (59%)

Fig. 7. Scanning Electron Micrographs of Fracture Surfaces of Haynes Alloy No. 25 Tensile Specimens. (a) After solution annealing. (b, c) After solution annealing and aging at 816°C.

Before presenting results on the fracture of Haynes alloy No. 25 in the 11,000-h-aged condition, we emphasize here several intrinsic features that contrast it with the annealed situation. First, aging riddled the alloy with a precipitate that formed semicontinuously in grain boundaries, rather continuously at twin boundaries, and well distributed as coarse particles and platelets throughout the grains. As remarked earlier, the secondary phase constituted 14.7% of the structure.

Secondly, because such a large amount of precipitate was formed, the matrix phase undoubtedly was substantially depleted of its elevated-temperature solute strengtheners, tungsten and chromium. A microprobe analysis of the matrix showed that it was depleted of tungsten by about 33 wt %, and that tungsten was uniformly distributed through the matrix. Bourgette¹⁰ also found matrix depletion (50%) in Haynes alloy No. 25 aged 1000 h at 870°C. This alloy depletion, no doubt, resulted in considerable softening of the matrix phase.

Finally, it is significant that, as a result of this matrix depletion, the alloy's capacity for precipitation was virtually spent, and, therefore, the alloy was more structurally stable.

An assessment of the experimental results for the 11,000-h-aged condition showed that pulling specimens below the minimum-ductility temperature produced fracture of an intergranular mode with little evidence of plasticity. This is illustrated in Fig. 5(d) for the specimen pulled at 649°C. Grains in the gage section showed meager extension and total elongation was only 15%. Figure 7(b) shows scanning electron micrographs of the fracture surface of the specimen pulled at room temperature. This specimen has basically the same failure mode as the 649°C specimen, although it is less ductile (10% total elongation). The topography is intergranular, and the granular texture seen on the faces projecting from the triple point (higher magnification) indicates brittleness and, further, may represent brittle phases.

Pulling the aged specimen at 760°C (the ductility minimum for the solution annealed condition), on the other hand, produced a transgranular fracture [Fig. 5(e)] and gave rise to the ductility inversion seen in Fig. 4. Grains in the gage section were extended, and total elongation measured 59%, as contrasted to only 21% exhibited by the solution annealed

specimen pulled at this temperature. Scanning electron micrographs of this fracture surface are shown in Fig. 7(c). The transgranular mode of the fracture is confirmed, and high ductility at surfaces is manifested by the many cusps along with several cup-and-cone separations. The more reflective surfaces in Fig. 7(b) and 7(c) may be twin boundaries.*

Aged specimens pulled above the minimum-ductility temperature also fractured transgranularly with good ductility, as illustrated in Fig. 5(f) for the test at 871°C. For the latter, total elongation was 62%.

That the aged specimens tested at the lower temperatures showed a lack of ductility was not surprising, since they contained the brittle Laves-Co₂W phase. Cracking quite understandably occurred at the grain boundaries, where the precipitate was largely strewn, except for an occasional cleavage at twin boundaries. That the improvement of ductility occurred immediately above the temperature at which recovery begins suggests that the underlying causes are thermally activated. It was first thought that stress relieving the tips of prevailing cracks was involved. However, separate experiments in which aged specimens were pulled at 760°C to a point just below the onset of unstable strain (Fig. 4) and then unloaded showed no prevailing cracks; the precipitate phases resisted cracking up to at least 35% elongation. This disproves the former and confirms something far more basic, namely, that the precipitates (Laves-Co₂W included) assume a more plastic character at this temperature. The microstructure of an aged specimen pulled at 760°C to 35% total elongation is illustrated in Fig. 8.

Another factor that probably contributed to the improvement in high-temperature ductility with aging at 816°C is the morphology of precipitates at the grain boundary. Whereas the grain boundary constituent was thin and virtually continuous in the solution annealed structure [Fig. 5(b)], it was coarse and semidispersed in the aged structure [Fig. 5(e)]. Such a dispersion would tend to resist the shearing at grain boundaries essential to wedge cracking. By intergranular cracking being averted,

* Such twin boundaries generally were lined with precipitates during aging and frequently cleaved cleanly at the lower test temperatures.

Fig. 8. Typical Crack-Free Microstructure of Haynes Alloy No. 25 Specimen Aged at 816°C and Pulled to 35% Total Elongation at 760°C. Section normal to gage. Arrow indicates stress axis. Etch: 100 ml HCl + 12 drops H₂O₂.

the aged specimen would be expected to undergo extensive transgranular deformation. This deformation, no doubt, is facilitated by a more plastic matrix softened by solute depletion. Only after extensive plastic deformation do microcracks form within the precipitate platelets (normal to the stress axis) and spread to the matrix. These matrix cracks are then blunted by local deformation. Also, pores form adjoining the rounder precipitate particles in the direction of the stress axis. Ultimately failure occurs transgranularly by fracture through the voids.

Evidence of the microcracks and pores formed in aged specimens pulled to failure at 649, 760, and 871°C are seen in the micrographs of Fig. 5(d-f). Crack blunting is prominent at 760°C [Fig. 5(e)], whereas pore formation at particle boundaries is common at 871°C [Fig. 5(f)].

The hardness curves of Fig. 6, interestingly, show a hardness inversion at the same temperature at which the ductility of the tensile

tests was reversed (Fig. 4). Below 660°C the pulled solution annealed specimens are harder than the aged specimens because the heavily worked solid-solution structure [Fig. 5(a)] predominates over the little-worked dispersion structure [Fig. 5(d)]; at 760°C and above the pulled aged specimens are the harder since the now heavily worked dispersion structure [Fig. 5(e) and (f)] predominates over the moderately worked structure relatively devoid of precipitates [Fig. 5(b) and (c)]. The hardness curves show a general hardness drop at the highest temperatures as a result of recovery.

Finally, it is important to note that the unstable portions of the strain (shaded regions of Fig. 4) in the specimens tested at 760°C were quite different for the two metal conditions. Whereas for the aged specimens the tensile-test curves were smooth throughout and the final fracture always occurred at about 80% or more of the ultimate stress, for the solution annealed specimens the flow curves were highly serrated at strains beyond the ultimate tensile stress (point of instability in Fig. 4), and fracture occurred at only a small fraction of the ultimate stress (less than 14% of it). This serration, which initiated in the solution annealed specimen at the onset of unstable strain, was linked with the ductility-minimum behavior and the intergranular cracking phenomenon that produces it. These serrations consisted of large yield drops to values substantially below the general level of the flow curve, which would then build up to successively lower stress levels with each drop. For the test represented in Fig. 4, fewer than 40 serrations accommodated the 86% total drop-off in stress level before final failure occurred.

Figure 9 illustrates the character of the intergranular cracking in its earliest stages at 760°C in solution annealed Haynes alloy No. 25. Figure 10 shows the nature of the cracking in the solution annealed specimen when pulled to failure at 871°C. In each of the micrographs, evidence is seen for the wedge-type voids¹¹ that form at triple junctions. The oval-type void¹² that forms along grain boundaries is also noted in Fig. 10.

Fig. 9. Intergranular Cracking in an Early Stage in Solution-Annealed Haynes Alloy No. 25 Pulled at 760°C. Section normal to gage. Arrow indicates stress axis, and x marks the wedge-type crack. As polished.

Fig. 10. Intergranular Cracking in Solution Annealed Haynes Alloy No. 25 Pulled at 871°C. Section normal to gage 13 mm from fracture surface. Arrow indicates stress axis. The X marks the wedge-type voids and Y the oval-type. Etch: 100 ml HCl + 12 drops H₂O₂.

DISCUSSION

The temperatures at which the minimum in ductility occurs as well as the magnitude of the dip are generally influenced by numerous variables, but chiefly by strain rate. Other important factors include the grain size, matrix hardening by solid solution or dispersion strengthening, and grain boundary precipitation. Sikka⁴ gives an excellent treatment of the effects of some of these variables on the minimum-ductility behavior in types 304 and 316 stainless steel. In our case a strain rate was chosen to accentuate the minimum and its reversal with prolonged aging at a single exposure temperature. We do not propose to elucidate the influence of interacting variables.

That aging improved the ductility at 760°C, the temperature of the ductility minimum in the unaged alloy, should not be taken to mean that aging treatments as a general rule have such effects. On the contrary, we should point out that the aging of thermally unstable alloys may produce an even greater deficiency in intermediate-temperature ductility if the prolonged aging is carried out at low temperatures. Examples of this follow:

Sikka recently showed for both mill annealed and solution annealed type 316 stainless steel⁴ a substantial improvement in ductility after prolonged aging at 649°C (the ductility-minimum temperature) and testing at the same temperature, but an equally imposing loss in ductility when long-time aging and testing were at 593°C (just below the minimum).

Also, Kimball et al.¹³ reported a severe loss in ductility of solution annealed alloy 625 as a result of prolonged aging in the 593 to 760°C (1100-1400°F) temperature range.* Yet we have shown in earlier work¹ that prolonged aging at a somewhat higher temperature, 816°C (minimum temperature for alloy 625), gives a high ductility.

* These authors attributed the loss in ductility to a matrix strengthening combined with the formation of continuous, brittle grain boundary precipitates.

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SUMMARY

Pronounced elevated-temperature tensile ductility minima occurred in solution annealed Haynes alloy No. 25 and Haynes alloy No. 188. The curves of total elongation versus test temperature inverted for these alloys after prolonged aging (11,000 h) at 816°C. The minima and their reversals were pronounced, occurring at approximately half the absolute melting temperature.

Haynes alloy No. 25, whose tensile properties were examined in some detail, in the solution annealed condition exhibited minimum behavior composed of three distinct regions. At temperatures up to the ductility minimum (760°C), deformation and fracture occurred by the usual transgranular crack-propagation mechanism, and total elongation was high. At the minimum temperature transgranular deformation was interrupted by intergranular cracking. Wedge-shaped voids produced at triple junctions by grain boundary shear grew unhindered, leading to intergranular failure and a reduction in total elongation. At temperatures above the minimum, fracture occurred by transgranular deformation acting in concert with grain boundary shear. Although failure still occurred by intergranular cracking, total elongation was high as a result of thermal recovery of crack fronts and the predominance of transgranular deformation.

After Haynes alloy No. 25 was aged for 11,000 h at 816°C, copious (14.7 wt %) precipitates, of which the Laves-Co₂W phase was dominant, formed at grain and twin boundaries and as coarse particles and platelets within grains. As a result, the matrix was depleted of solute hardeners (tungsten reduced by 33%) and was significantly softened.

At test temperatures up to 760°C, corresponding to the ductility minimum for the solution annealed condition, early fracture of the aged tensile specimens was induced by the fragile precipitates, and failure was intergranular. Total elongation, understandably, was low. At 760°C, in contrast to the solution annealed condition, fracture occurred transgranularly, and total elongation was again high. This renewal of ductility, which persisted to still higher temperatures, was attributed to several contributing factors: (1) The brittle precipitate phases toughened at 760°C and resisted cracking. (2) Precipitate particles

dispersed about the grain boundaries inhibited intergranular cracking by thwarting grain boundary shear. (3) Transgranular deformation was enhanced by a more plastic matrix softened by solute depletion. (4) Cracks, once formed, were blunted by local deformation and thermal recovery.

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