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# DUCTILITY MINIMUM AND ITS REVERSAL WITH PROLONGED AGING IN COBALT- AND NICKEL-BASE SUPERALLOYS<sup>1</sup>

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## ABSTRACT

The cobalz-base superalloys Haynes alloy No. 25 and Haynes alloy No. 188 and the nickel-base superalloy Inconel 625 show a pronounced ductility minimum at temperatures of 760 and 704 °C, respectively, in the solution-annealed condition. However, after prolonged (11,000 h) aging at 816 °C (1500 °F), copious precipitates form to completely reverse the behavior. These precipitates reduce tensile ductility drastically up to temperatures where the ductility dip is observed for the solution-annealed condition; then the brittle behavior from aging gives way to greatly "nhanced ductility. This behavior in Haynes alloy No. 25 is examined in detail. Its 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, which began with the onset of recovery.

#### INTRODUCTION

mong the various criteria for evaluating high-temperature alloys for nuclear and solar energy-conversion applications, good mechanical properties and their retention in the face of prolonged thermal aging rarks high. In connection with a broad study of superalloys for such applications — the study presently includes more thermally stable Hastelloy S, Inconel 617, and Inconel 618 — we examined temperature dependence of the tensile properties of several of the earlier superalloys. Particular attention was given the intermediate-temperature ductility loss that occurs in the solution-annealed condition and the effects of prolonged aging at high temperatures: 11,000 h at  $816^{\circ}C$  (1500°F) (1).

For the alloys examined this aging exposure precipitated substantial amounts of carbide and intermetallic phases. These precipitates, as expected, promot i severe embrittlement at low test temperatures; but at the intermediate temperature  $[0.5-0.6\ T_m$  (1033 K)], where the ductility minimum ordinarily occurs for solution-annealed material, ductility was restored.

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To better understand these phenomena, we studied Haynes alloy No. 25 in some detail.

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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 strain rates as in creep (5).

# EXPERIMENTAL

The investigation was conducted on three heats of Haynes alloy No. 25  $(0.0_{-}, 0.11, \text{ and } 0.35\% \text{ Si})$ ,<sup>2</sup> and one heat each of Haynes alloy No. 188 and Inconel 625. The chemical composition of each of the various heats (1) conformed to the corresponding materials specifications (AMS 5537C, AMS 5608, and ASTM B443-75, respectively). Haynes alloy No. 25 and Haynes alloy No. 188 are cobalt base, 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 an article by Herchenroeder et al. (7) describes precipitation in Haynes alloy No. 188. Inconel 625 is a nickel-base alloy containing 22 wt % Cr, 9 wt % Mo, and 3.5 wt % Nb + Ta as important alloying additions. The precipitation hardening in this alloy is treated by Kimball et al. (8).

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. The long-term (11,000-h) aging was performed in vacuum on machined specimens given a solution anneal beforehand (1). 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 0. in./min in a 44.5-kN (10, NO0-1b) Instron tensile testing machine. The detail d tensile data gathered on the different alloys have been documented (1).

In the present work extensive studies were made of the deformation and aging responses of the specimens of Haynes alloy No. 25 tested both as solution annealed and after 11,000-h aging at 816°C. These studies included examinations by optical and scanning electron metallography, microhardness tests, x-ray diffraction, and x-ray fluorescence analysis of matrix depletion.

### RESULTS

The yield strength, ultimate tensile strength, and total elongation determined previously as a function of test temperature are shown as composite graphs for two of the Haynes alloy No. 25 heats, Haynes alloy No. 188, and Inconel alloy 625 in Figs. 1, 2, and 3, respectively. The solution-annealed and 11,000-h-aged conditions are included for comparison. 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.

Observe that for all the alloys the 11,000-h aging results in poor ductility at room temperature and 316°C compared with results for the solution-annealed condition, but that at 760°C ductility is restored. Significantly, this restoration of ductility occurs in the temperature region where ductility for the solution-annealed condition achieves its minimum, thus prompting the term "minimum reversal." At 982°C, a temperature where ductility for the solution-annealed condition is well restored, aged ductility remains high. It is noteworthy that while 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. The exception to this is the comparatively high yield strength recorded for Inconel 625 at room temperature to 760°C in the aged condition (Fig. 3).

<sup>2</sup>Heats with varying silicon content were included, as silicon may accelerate precipitation with aging. However, silicon proved to have little or no effect for the aging conditions we employed.

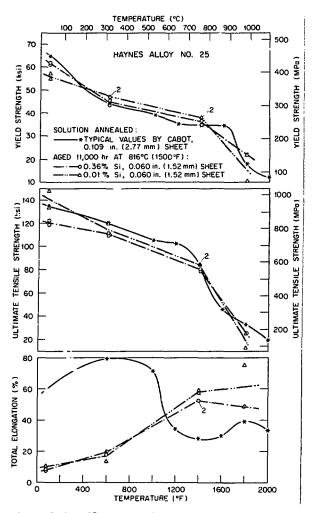


Fig. 1 Comparison of the effects of solution annealing with those of aging at 816°C on yield strength, ultimate tensile strength, and total elongation of Haynes alloy No. 25. Data on solution-annealed specimens are from Haynes alloy No. 25, brochure F-30,041E, Cabot Corporation, Kokomo, Ind., 1970.

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 parameter is a product of strength and ductility. In Fig. 4 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 reversal in the 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 ductilityminimum temperature) and greatly exceeds that for the solution-annealed

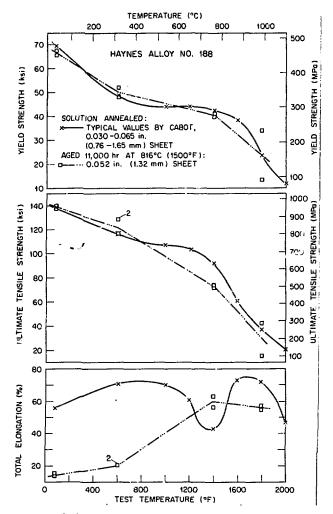


Fig. 2 Comparison of the effects of solution annealing with those of aging at 816°C on yield strength, ultimate tensile strength, and total elongation of Haynes alloy No. 188. Data on solution-annealed specimens are from Haynes alloy No. 188, brochure F-30,361B, Cabot Corporation, Kokomo, Ind., 1973.

condition at this temperature (1). The large dropoff 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 in the solution-annealed condition and the reversal the alloy experiences with 11,000-h aging at 816°C (1500°F).

Rhines and Wray (9) 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 and alloys. 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 dropoff of total elongation

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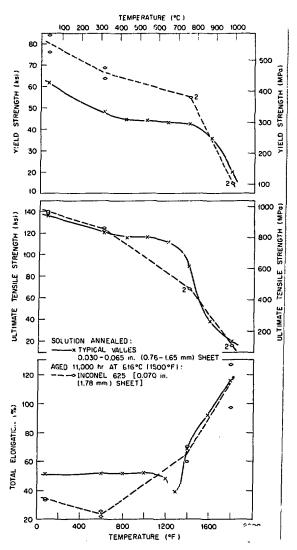
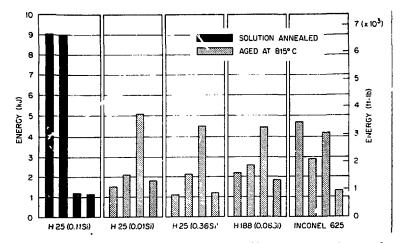


Fig. 3 Comparison of the effects of solution annealing with those of aging at 816°C on the yield strength, ultimate tensile strength, and total elongation of Inconel 625. Data on solution-annealed specimens were obtained from private communication, Charles Spenaugle, Huntington Alloy, International Nickel Company, Huntington, W. Va., April 1976.

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 (9). Thus, ductility increases again. In reporting findings of



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Fig. 4 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.

high-strain fracture in Inconel aloy 600 resulting from hot torsion testing, Shapiro and Dieter (2) concurred in the generalized theory advanced by Rhines and Wray. Later, Arkoosh and Foire (10) examined ductility-minimum behavior in Hastelloy alloy X (a nickel-base superalloy) and confirmed the theory of Rhines and Wray, with one exception. They contended that restoration of ductility above the winimum results from matrix depletion and that recrystallization is not realized until much higher temperatures are reached.

Whereas the foregoing concepts have been advanced to explain the ductility-minimum phenomenon so prevalent in metal and alloys, little appears in the literature on the reversal effect. Figure 5 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 marking the onset of unstalle station (defined here as elongation at maximum true stress) are included. Significantly, this graph shows that the improvement in ductility at the minimum resulting from 11,000-h aging is largely stable strain.

In explaining the underlying principles of this graph it is expedient to treat 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 to intergranular for the solution-annealed condition and from intergranular to transgranular for the 11,000-h-aged condition with rising temperature in this range. Generally transgranular failure involved large total giongation, as manifested by an extension of grains in the loading direction. On the other herd, intergranular 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.

Photomicrographs illustrating the microstructure at the fracture faces for the various solution-annealed and 11,000-h-aged specimens pulled in this transition region are shown in Fig. 6. 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 solutionannealed and the 1 000-h-aged conditions. Curves of hardness at room temperature versus in perature during tensile testing of the Haynes alloy No. 25 are compared for the two conditions in Fig. 7.

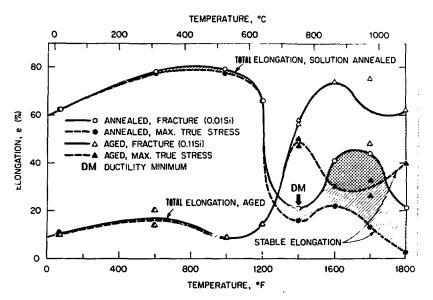


Fig. 5 Stable and total elongation of Haynes alloy No. 25 as influenced by metal condition and test temperature.

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A final aid to interpreting the results is the following information excerpted from the work of Yukawa and Satu (6) on the identification and kinetics of phase precipitation in the Haynes alloy No. 25. The aging process proposed at 800 °C (1472 °F) is:

$$(M_7C_3) \div M_{23}C_6 \nrightarrow M_6C \nrightarrow Laves - Co_2W \nrightarrow \mu - Co_7W_6$$
(1)

The phase in parenthesis is metastable. Age hardening at 800°C and higher is attributed to precipitation of carbides (M<sub>23</sub>C<sub>6</sub> and M<sub>6</sub>C) and Laves-Co<sub>2</sub>W. At temperatures below 800°C, 100 h and longer is required to initiate precipitation of the Laves-Co<sub>2</sub>W phase, whereas the carbides are formed in much shorter periods. The Laves-Co<sub>2</sub>W phase is rather fragile and is thought to promote brittleness in Haynes alloy No. 25 (11), whereas the carbide phases generally are tougher. For our aged Haynes alloy No. 25 specimens, a weighing and an analysis by x-ray difficaction of residues extracted by electrolysis indicated a total of 14.7 wt % precipitate had formed; approximately half of this was Laves-Co<sub>2</sub>W and the remainder unidentified phases, perhaps  $\mu$ -Co $_7W_6$ 

As in "evious findings on ductility-minimum behavior (2,9,10), we found fracture in the solution-treated alloys to occur by the usual transgranular crack propagation mechanisms at temperatures below the transition (649°C and lower), and elongation, accordingly, was very high. This will be noted from the microstructure after testing at 649°C (Fig. 6a). Total elongation was 66%, and grains were extended along the gage section.

At the minimum-ductility temperature  $(760 \,^{\circ}\text{C})$  intergranular voids formed at triple junctions as a result of grain boundary shear and eventually grew unimpeded to give early intergranular fracture (Fig. 6b). 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. 8a that failure indeed is intergranular, but the distinctive cusps seen at higher magnification indicate high localized plasticity. Similar features prompted Rhines (9) to coin the expression "plastic brittleness" for this behavior.

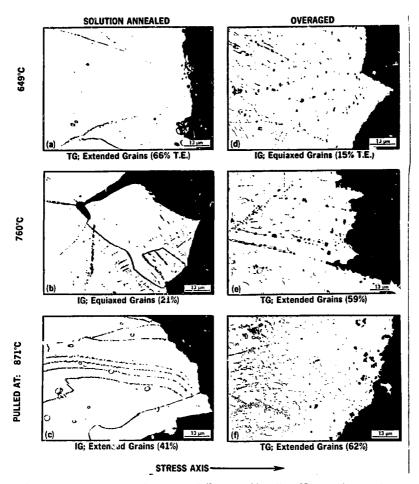


Fig. 6 Microstructures at fracture of Haynes alloy No. 25 tensile specimens pulled either after solution annealing or after solution annealing and aging at 816°C. Etchant: 100 ml HCl + 12 drops  $\rm H_{2}O_{2}$ .

The minimum behavior here proved so pronounced that we suspected that strain-induced submicroscopic precipitation hardening (by carbides) and concommitant softening near boundaries by denudation abbetted the cracking. However, the low hardness exhibited by the specimen displaying the minimum ductility in Fig. 7 (solution annealed, pulled at 760 °C) suggests that this may not be the case. Instead, the intergranular cracking may be aided by the dynamic dislocation recovery mechanism postulated by Sikka (4). He suggests that work-hardened grains are preferentially recovered adjacent to grain boundaries as a result of a low vacancy diffusion rate and, as a consequence, cavitation cracking is facilitated.

At 871°C fracture occurred as a result of predominantly transgranular deformation acting in concert with intergranular cracking (Fig. 6c). 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,

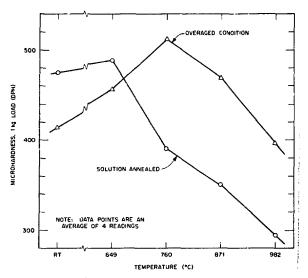


Fig. 7 Comparison of microhardness values at room temperature after pulling solution-annealed and overaged specimens at various temperatures.

dislocation entanglements throughout grains are expected to recover as dislocations climb to form substructures and, on occasion, are annihilated. Also, intergranular cracking is delayed by thermal recovery of crack fronts. Unlike the findings of Rhines  $(\underline{9})$ , evidence for recrystallization isolating intergranular cracking and in this way enhancing ductility was lacking.

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

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 precipitates that formed semicontinuously in grain boundaries, rather continuously at twin boundaries, and were well distributed as small particles and platelets throughout the grains. As we remarked earlier, the secondary phase constituted 14.7 wt % 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. Bourgette (11) also found matrix depletion (50%) in Haynes alle  $\pm$  Bo. 25 aged 1000 h at 870 °C. This alloy depletion, no doubt, resulted in contailerable 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. 6d for the specimen pulled at  $649^{\circ}$ C. Grains in the gage section showed meager extension and total elongation was only 15%. Figure 8b shows scanning electron micrographs of the fracture surface of the specimen pulled at room temperature, which has basically the same failure mode as the former specimen although it is less ductile (10% total elongation). The

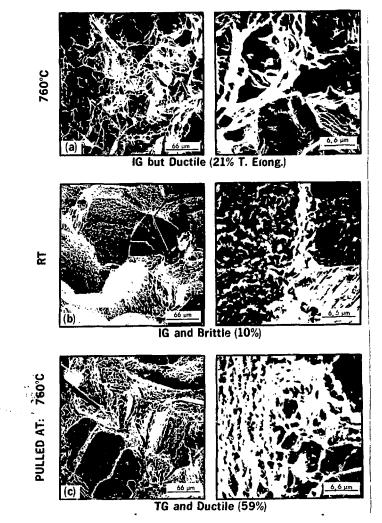


Fig. 8 Scanning electron micrographs of fracture surfaces of Haynes alloy No. 25 tensile specimens. (a) After solution annealing. (b),(c) After solution annealing and aging at 816 °C.

topology is intergranular, and the granular texture seen on the faces projecting from the triple point (higher magnification) indicates brittleness and, furthermore, may represent brittle plases. Pulling the aged specimen at 760°C (the ductility minimum for the

Pulling the aged specimen at 760°C (the ductility minimum for the solution-annealed condition), on the other hand, produced a transgranular mode of fracture (Fig. 6e) and gave right to the dramatic ductility inversion seen in Fig. 5. Grains in the gage section were extended and total elongation measured 59%, as contrasted to only 21% experienced by the solution-annealed specimen pulled at this temperature. The scanning electron micrographs of this fracture surface are shown in Fig. 8c. The transgranular mode of the fracture is confirmed, and high ductility at surfaces 's mar ifested by the many cusps along with several cup-and-cone type separations. The more reflective surfaces in Fig. 8 (b and c) may be twin boundaries.<sup>3</sup>

Fracture of aged specimens pulled above the minimum-ductility temperature also occurred transgranularly with good ductility, as illustrated in Fig. 6(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 the Laves-Go2W phase they contained was brittle. Cracking quite understandably occurred at the grain boundaries where the precipitate was largely strewn, except for an occasional cleavage at twin boundaries. That the inversion of ductility occurred immediately above the temperature where recovery begins suggests that the underlying causes are thermally activated. It was first thought that stress-relieving the tips of prevaiing cracks was involved. However, separate experiments in which aged specimens were pulled at 760°C to a point just below the precipitate phases resist cracking up to very high levels of extension. 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 overaged specimen pulled at 760°C to 35% total elongation is illustrated in Fig. 9.

Another factor that undoubtedly contributed to the reversal in ductility with aging at  $816^{\circ}C$  is the change in the morphology of precipitates at the grain boundary. Whereas the grain boundary constituent was thin and virtually continuous in the solution-annealed structure [Fig. 6(b)], it was coarse and semidispersed in the aged structure [Fig. 6(e)]. Such a dispersion would tend to resist the shearing at grain boundaries essential to cavitation cracking.

<sup>3</sup>Such twin boundaries generally were lined with precipitates during overaging, and frequently cleaved cleanly at the lower test temperatures.



Fig. 9 Typical crack-free microstructure of Haynes alloy No. 25 spec<sup>+</sup> ged at 816°C and pulled to 35% total elongation at 760°C. Section norm. , age. Arrow indicates stress axis. Etchant: 100 ml HCl + 12 drops H<sub>2</sub>O<sub>2</sub>.

By having averted early intergranular cracking, 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.

In the final stages of deformation, microcracks formed normal to the stress axis within the precipitates (platelets) and spread to the matrix. The matrix cracks were then blunted by local deformation. Pores formed adjoining the rounder precipitate particles in prolongation with the stress axis. Ultimately, failure occurred transgranularly by fracture through the voids.

Evidence of the microcracks and pores formed in aged specimens pulled at 649, 760, and 871°C are seen in the micrographs of Fig. 6(d-f). Crack blunting is prominent at 760°C [Fig. 6(e)], whereas pore formation at particle boundaries is common at 871°C [Fig. 6(f)].

The hardness curves of Fig. 7, interestingly, show a hardness inversion to occur at the same temperature where the ductility of the tensile tests was reversed (Fig. 5). Below 760°C the pulled solution-annealed specimens are harder than the aged specimens because the heavily worked solid solution structure [Fig. 6(a)] predominates over the little-worked dispersion structure [Fig. 6(d)]; at 760°C and above the pulled aged specimens are the harder since the now heavily worked dispersion structure [Fig. 6 (e and f)] predominates over the moderately worked structure relatively devoid of precipitates [Fig. 6 (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. 5) 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. 5), and fracture occurred at only a small fraction of the ultimate tensile stress (less than 14% of it). This serrated effect, initiated in the solution-annealed specimen at the onset of unstable strain, was linked to 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. 5, fewer than 40 serrations accommodated the 86% total dropoff in stress level before final failure occurred.

Figure 10 illustrates the character of the intergranular cracking in its earliest stages at  $760^{\circ}$ C in solution-annealed Haynes alloy No. 25. Figure 11 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 void (12) that forms at triple junctions. The oval-type void (13) that forms along grain boundaries is noted in the latter micrograph.

### 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. Sikka (4) 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 study a combination of strain rate and temperature 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 we obtained so much ductility at the minimum temperature should not be taken to mean that aging treatments generally have such an effect. On the contrary, we should point out that the aging of thermally unstable alloys may produce an even greater deficiency in intermediate-temperature ductility where the prolonged aging is carried out at low temperatures. Examples of this are the following.

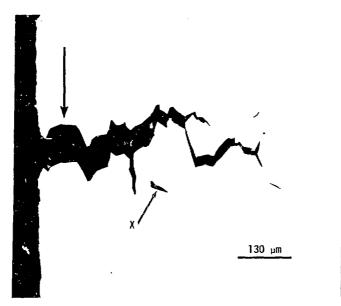


Fig. 10 Intergranular cracking in an early stage in solution-annealed Haynes alloy No. 25 pulled at 760°C. Section normal to gage. Arrow indicates stress axis whereas X marks the wedge-type crack. As polished.

Sikka recently showed for both mill-annealed and solution-annealed type 316 stainless steel (4) 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 at 593 °C (just below the minimum).

Also, Kimbal et al. reported a severe loss in ductility of solutionannealed Inconel 625 (8) as a result of prolonged aging in the  $593-760^{\circ}C$ (1100-1400°F) temperature range.<sup>4</sup> Yet we have shown that prolonged aging at a somewhat higher temperature,  $816^{\circ}C$ , gives the high ductility at the minimum-ductility temperature.

### SUMMARY

Pronounced tensile ductility minima occurred in solution-annealed Haynes alloy No. 25, Haynes alloy No. 188, and Inconel 625. Inversions of the total elongation-test temperature curves occurred 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, exhibited minimum behavior in the solution-annealed condition composed of three distinct regions. At temperatures up to the ductility minimum (760°C), deformation and fracture occurred by the usual transgranular crackpropagation mechanism and total elongation was very 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 drastic reduction in total elongation. At temperatures above the minimum, fracture occurred by transgranular deformation acting in concert with grain boundary shear. Although

<sup>&</sup>quot;These authors attributed the loss in ductility to a matrix strengthening combined with the formation of continuous, brittle grain boundary precipitates.

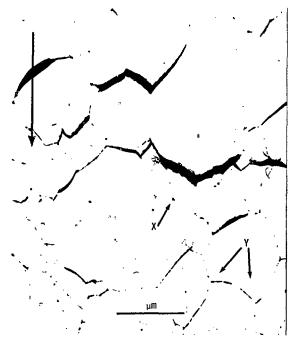


Fig. 11 Intergranular cracking in solution annealed Haynes alloy No. 25 pulled at 871°C. Section normal to gage 1/2-in. removed from fracture surface. Arrow indicates stress axis. The X marks the wedge-type void and Y the oval-type. Etchant: 100 ml HCl + 12 drops  $H_2O_2$ .

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 aging Haynes alloy No. 25 for 11,000 h at 816°C, copious (14.7 wt %) precipitates, of which the Laves-Co<sub>2</sub>W phase was dominant, formed at grain and twin boundaries and as well distributed bulky particles and platelets within the grains. As a result the matrix was depleted of solute hardeners (tungsten reduced by 33%) and was significantly softened.

At test temperatures up to  $760\,^{\circ}$ 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 very low. At the minimum, in contrast to the solution-treated condition, fracture occurred transgranularly, and total elongation was again high. This reversal in ductility, which persisted to still higher temperatures, was attributed to several contributing factors: (1) the brittle precipitate phases toughened at  $760\,^{\circ}$ 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, and (4) cracks, once formed, were blunted by local deformation and thermal recovery.

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## REFERENCES

1 Hammond, J. P., "Effect of Long-Term Aging at 815°C on the Tensile Properties and Microstructural Stability of Four Cobalt- and Nickel-Base Superalloys," ORNL-5174, Aug. 1976, Oak Ridge National Laboratory, Oak Ridge, Tenn.

2 Shapiro, E. and Dieter, G. E., "High Temperature High Strain Rate Fracture of Inconel 600," Metallurgical Transactions, Vol. 1, No. 6, June 1970, pp. 1711-1719.

3 Duvall, D. S. and Owezarski, W. A., "Studies of Postweld Heat-Treatment Cracking in Nickel-Base Alloys," Welding Journal, Vol. 48, No. 1, 1969, pp. 105-225.

 4 Sikka, V. K., "Elevated Temperature Ductility of Types 304 and 316
 Stainless Steel," found elsewhere in this proceedings.
 5 Savage, W. F., and Krantz, B. M., "Microsegregated in Autogeneous
 Hastelloy X Weld," Welding Journal, Vol. 50, No. 7, 1971, pp. 292--303-s. 6 Yukawa, N. and Sato, K., "The Correlation between Microstructure and

Stress Rupture Properties of a Co-Cr-Ni-W (HS-25) Alloy," Proceedings of the International Conference on the Strength of Metals and Alloys, The Japan Institute of Metals, Omachi, Sendai, Japan, 1968, pp. 680-686.

7 Herchenroeder, R. B., Matthews, S. J., Tackett, J. W., and Wlodek,
 S. I., "Haynes Alloy No. 188," *Cobalt*, No. 54, March 1972, pp. 3-13.
 8 Kimball, O. F., Pieren, W. R., and Roberts, D. R., "Effects of Elevated-

Temperature Aging on the Mechanical Properties and Ductility of Ni-Cr-Mo-Cb Alloy 625," GULG-GA-Al2683, Oct. 1973, General Atomic Company, San Diego, Calif.

9 Rhines, F. N., and Wray, P. J., "Investigation of the Intermediate Temperature Ductility Minimum in Metals," *Transactions of the American Society* for Metals, Vol. 54, 1961, pp. 117-128.

10 Arkoosh, M. A., and Fiore, N. F., "Elevated Temperature Ductility Minimum in Hastelloy Alloy X," Metallurgical Transactions, Vol. 3, No. 9, Aug. 1972, pp. 2235-2240.

11 Bourgette, D. L., "Effect of Aging Time and Temperature on the Impact and Tersile Behavior of L-605 - A Cobalt-Base Alloy," ORNL-TM-3738, April 1973, Oak Ridge National Laboratory, Oak Ridge, Tenn.

1

12 Zener, C., "The Micro-Mechanism of Fracture," Fracturing of Metals, American Society for Meta s, Cleveland, 0., 1947, p. 3. 13 Chen, C. W., and Machlin E. S., "Letters to the Editor," Acta

Metallurgica, Vol. 4, No. 6, 1956, p. 655.