# 26. Intercrystalline Failure at High Temperatures

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#### ABSTRACT

This chapter presents a discussion of the influence of composition and testing variables on the incidence of intercrystalline fracture. Experimental results are presented that illustrate the effect of deformation, test temperature, and stress on the extent of grain-boundary sliding in aluminum alloys. Theories of intercrystalline fracture and the role of grain-boundary sliding, void formation, grain-boundary migration, and dislocation pile-ups are assessed.

## Introduction

Changes in temperature or in strain rate (by a factor of 10 or more) have but a small effect in the low-temperature testing and evaluation of materials that fail by ductile fracture. In contrast, testing at high temperatures shows that strength, ductility, and deformation and fracture characteristics are strongly affected by temperature, strain rate, grain size, prior history, and even the atmosphere in which the tests are performed. Whereas one correctly assumes that a structure tested at low temperature is generally stable, this can never be the assumption at high temperatures. Not only are there changes in structure (such as aging or overaging), but there are such changes as surface oxidation, grain growth, and recrystallization.

Under these circumstances, the test specimen used to initiate the test is difficult to recognize as the same material shortly thereafter. In high-temperature testing, it is not unusual to have tests under way for periods of time up to several thousand hours.

For these reasons, one must define the test conditions quite precisely in order to establish the significance of the data.

At elevated temperatures (relative to the melting temperature), when

temperature increases and strain rates decrease, grain-boundary deformation processes become progressively more important. With decreasing strain rate, fractures tend to change from the transcrystalline variety to intercrystalline; intercrystalline cracking becomes the ultimate failure mode and is accompanied by decreasing ductility, which often leads to apparent brittleness. The rate of change (as a function of strain rate) from transcrystalline to intercrystalline failure is a function of such factors as the alloy composition, the structure, cleanliness, and grain size. The change is more rapid but clear-cut in complex alloys, while it is quite slow, complex, and involved in pure metals and simple alloys because of the more extensive grain-boundary migration in the latter.

For these reasons, and as will be illustrated below, it is difficult to define the test conditions desired for a study of high-temperature fracture. In particular, because of the high temperatures involved, the structure itself is subject to change, and this further complicates the fixing of all test variables.

For example, even the definition of the term "ductility" is a difficult problem. Reduction-in-area values do not conform to total-elongation values in many systems, primarily because much of the elongation occurs in third-stage creep and is associated with the opening of intercrystalline

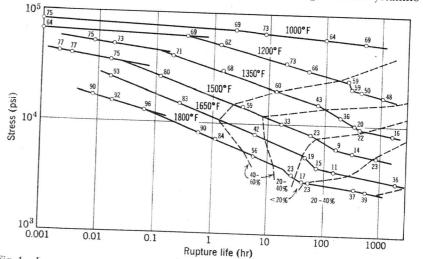


Fig. 1. Log stress versus log rupture life plot of vacuum-melted 80 nickel-20 chromium alloy containing 1.33% silicon. Dashed curves delineate reduction-in-area zones.

cracks. Reduction-in-area values are much less affected by crack enlargement (Fig. 3). Figure 1 shows a log stress-log rupture-time plot for a vacuum-melted 80 nickel-20 chromium-type alloy containing 1.33%

silicon, which illustrates the change in reduction in area as a function of temperature and rupture time.<sup>1</sup> Intercrystalline cracking is first noted at approximately the first break in the curves at 1000° to 1500°F and at all test stresses at 1650° and 1800°F. Such cracking becomes more severe with decreasing stress and results in intercrystalline fracture at a stress somewhat lower than that indicated at the first break.

Figure 2 shows the elongation values for this alloy (vacuum melted) and a comparable air-melted alloy at 1200°, 1500°, and 1800°F. The

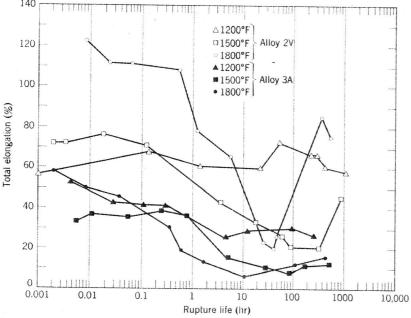


Fig. 2. Total elongation as a function of log rupture time and temperature for vacuum-melted (2V) and air-melted (3A) 80 nickel-20 chromium alloys containing about 1.3% silicon.

large decrease in ductility is readily evident for both alloys at 1500° and 1800°F.

Figure 2 is of particular interest because it shows an increase in ductility in the long-time tests at 1500° and 1800°F, the increase being associated with large atmospheric strengthening effects (grain-boundary oxidation, internal oxidation, nitrogen solution). The fractures continue to be of the intercrystalline type. Similar longer time tests in argon do not show the ductility increase noted in Fig. 2, but they also do not change the type of fracture.<sup>2</sup> The long-time tests in argon are significantly weaker. Note that the ductility behavior of the air-melted alloy is similar to that

of the vacuum-melted alloy but that the ductility values under all circumstances are one-half to one-third those of the vacuum alloy.

Figure 3 compares the fracture appearance of the air- and vacuum-

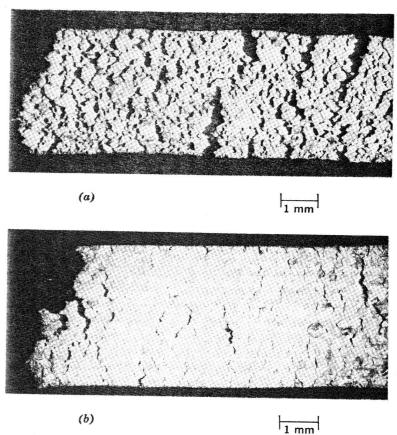


Fig. 3. (a) Vacuum-melted 80 nickel-20 chromium alloy containing 1.33% silicon tested at 1500°F, 5000 psi. Rupture life, 870 hr; clongation, 45%. (b) Air-melted alloy also tested at 1500°F, 5000 psi. Rupture life, 524 hr; clongation, 12%.

melted 80 nickel-20 chromium alloys, both tested in air at 1500°F. There is a marked difference not only in the size and number of intercrystalline cracks, but also in their distribution and continuity. Since the compositions are otherwise similar, Figs. 2 and 3 illustrate the large effect of minor elements and impurities on the observed fracture propagation of such an alloy.

Table 1 shows that the vacuum-melted alloy not only has greater total elongation, but shows greater ductility even after subtraction of the

fictitious elongation attributable to the opening up of intercrystalline  ${\rm cracks.}^2$ 

TABLE 1. Contribution of Crack Widening to Total Elongation at 5000-psi Stress

Alloy	Temperature (°F)	Total Elongation at Fracture $E_{\text{tot}}$ (%)	Elongation Attributable to Cracks $E_e$ $(\%)$	$E_{\text{tot}} - E_{\text{(%)}}$
2V*	1500	45	18	27
2V	1500	12	3.5	8.5
3.A.	1800	56	17	39
~ A A	1800	16	9	7

<sup>\*</sup> V = vacuum-melted; A = air-melted

In contrast to the behavior of these commercial, but simple, singlephase alloys, the behavior of pure metals and very pure simple alloys is more complex and difficult to define.

Thus we find that in high-purity aluminum (99.994%), even though high-temperature deformation processes such as grain-boundary sliding and grain-boundary migration are operative, fractures are wholly ductile and transgranular to the melting temperature.3 Voids at grain boundaries are not observed or are rare, and slip remains the primary mode of deformation to the melting temperature. However, high-purity solidsolution alloys of aluminum containing copper, magnesium, or zinc, when tested at 500° to 600°F, do show intercrystalline cracking and abundant voids at the grain boundaries, these occurrences being associated with a loss of ductility. Intercrystalline fracture also occurs only in a small temperature-strain rate region.4,5 An increase of test temperature from 500° to 700°F (and higher) results in elimination of intercrystalline cracking and failure as more extensive grain-boundary migration, comparable to that noted in high-purity aluminum, comes into play (along with other high-temperature processes), and ductility is not only recovered but attains very high values.

Further increases in alloy content or in impurity level expand the temperature-strain rate region in which intercrystalline cracking occurs, accompanied by still greater losses in ductility.

In some alloys, even a few parts per million or a few thousandths of a percent of particular impurities are adequate to bring about intercrystalline cracking with large or almost total loss of ductility at fracture in hot creep.

These remarks are intended to point up not only the variations in hot fracture as a result of test conditions, but also the variations resulting from alloy preparation. Concern must exist for both the initiation of intercrystalline cracking and for the mechanism of crack propagation, because the incidence of intercrystalline cracking is not a guarantee of intercrystalline failure.

# Deformation Prior to Intercrystalline Cracking

If one examines how the grain boundary is behaving during hot creep, in comparison to the behavior of the grains, the following observations can be made.

1. The contribution of grain-boundary sliding to total creep is greatest at small strains; the ratio  $E_{\rm gb}/E_{\rm tot}$  reaches a constant value of about 9 to 11% after about 5 to 10% strain (Fig. 4). In these aluminum alloys,

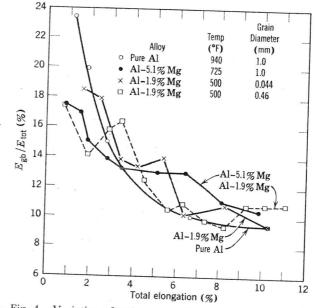


Fig. 4. Variation of ratio of grain-boundary sliding to total elongation as a function of creep strain for aluminum and several solid-solution alloys from 500° to 940°F and for several grain sizes.

neither composition, grain size, nor temperature appears to affect the amount of grain-boundary sliding significantly.<sup>6</sup> During the course of creep, at strains of less than about 5%, there appears to be constant

readjustment of the amount of grain and grain-boundary shear such that the above ratio is never too far from that of an average band, as indicated in Fig. 4. In such ductile materials, this would indicate a defense against excessive sliding, even on those boundaries most favorably oriented for shear, without constant deformation adjustment in the grains. Such behavior is a means of avoiding extensive early intercrystalline cracking. Of the materials shown in Fig. 4, the two alloy compositions showed intercrystalline cracking but transgranular failure, and the pure aluminum showed only ductile transgranular fracture.

2. The ratio  $E_{\rm gb}/E_{\rm tot}$  is very small below 400°F and must soon thereafter approach zero. Above 400°F, this ratio reaches a maximum of

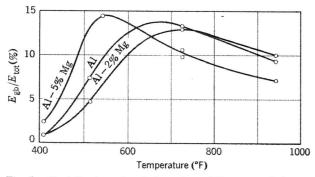


Fig. 5. Contribution of grain-boundary sliding to total elongation, in percent, for pure aluminum and two alloys, plotted as a function of temperature, after approximately 10% strain.

about 14% for aluminum alloys, the maximum being achieved between about 500° to 700°F. After this, there is a slight decrease at higher temperatures (Fig. 5). These values <sup>6</sup> are lower than those reported by others <sup>7,8</sup> but have been shown to be more correct.

While the above ratio remains relatively constant between about 10 and 12% at the higher temperatures, the behavior of the grain boundary hardly stays constant. In aluminum alloys, with temperature increasing above about 400°F, the ease of grain-boundary migration increases rapidly and becomes progressively more extensive. At about 500°F, total grain-boundary sliding is extensive, but boundaries migrate to unstrained positions slowly, permitting accumulation of strain energy in narrow bands near the grain boundary. With increasing temperature, grain-boundary sliding occurs in smaller, more frequent intervals, with migration occurring much more readily. As a result, fracture characteristics also change rapidly.

3. The ratio  $E_{\rm gb}/E_{\rm tot}$  increases with decreasing stress, but after reach-

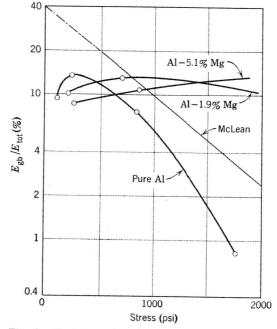


Fig. 6. Variation of  $E_{\rm gb}/E_{\rm tot}$  as a function of stress. Note position of data by McLean.<sup>7</sup>

ing a maximum at low stress values, it decreases as the stress approaches zero <sup>6</sup> (Fig. 6).

## Void Formation in Intercrystalline Cracking

It was suggested by Greenwood 9 that vacancies flow to regions of high tensile stress and condense into voids at grain boundaries normal to the stress. These voids, by subsequent linking, are thought to lead eventually to intercrystalline failure. Greenwood, Miller, and Suiter 10 made the observation that the first stages of intercrystalline cracking were associated with the increase and growth of the voids, the roughened irregular intercrystalline crack surfaces serving as evidence that the linking of voids leads to fracture.

Gifkins <sup>11</sup> suggested that grain-boundary jogs were formed because of slip, the size of the jog being proportional to the number of dislocations passing across the boundary to initiate slip in the adjoining grain. The pile-up of dislocations at the jog was thought to result in a stress field that could lead to intercrystalline fracture if the boundary does not migrate. Lack of voids in pure aluminum is thus attributed to easy

migration of the boundary at any temperature at which grain-boundary shear takes place.

Chen and Machlin <sup>12</sup> and Chen <sup>13</sup> concluded that grain-boundary sliding is a prerequisite for intercrystalline cracking. With such grain-boundary shear, the stresses that can result at jogs can readily produce fracture.

Crussard and Friedel <sup>14</sup> indicated that in an elliptical hole under creep conditions the net effect of vacancy and atom movements would be to cause propagation of the hole to form a crack; however, Friedel <sup>15</sup> subsequently was reported to be of the opinion that vacancies would be attracted to the major axis of the elliptical hole because of the high shear stress at the tips of the axis. Either mechanism would have the net effect of increasing crack propagation. Calculations by Machlin <sup>16</sup> indicated that vacancy condensation could not nucleate voids but that submicroscopic voids are already present in the metal as faults of casting or recrystallization.

# Recent Test Observations of Intercrystalline Cracking

Grain-boundary shear has been observed at temperatures as low as room temperature for high-purity aluminum and an aluminum-zinc alloy <sup>17,18</sup> (about 0.33 of the absolute melting temperature). More extensive grain-boundary sliding than that occurring at room temperature has been observed at 400°F (Fig. 5) for both pure aluminum and several solid-solution alloys. In neither of these instances was void formation observed,<sup>6</sup> and fractures were transcrystalline. Thus some grain-boundary sliding is of itself no guarantee either of void formation or intercrystalline cracking. Even the absence of significant boundary migration at these lower temperatures does not significantly promote void formation, and fractures are typically ductile, with slip the major deformation mechanism.

For conditions of comparable grain size, strain rate, temperature, and equivalent grain-boundary shear, we note from Figs. 4 and 5 and allied experimental observations that:

- (a) High-purity aluminum does not show voids and fails by transgranular means at all temperatures.
- (b) In the leaner solid-solution alloys, such as the 2% magnesium alloy, voids are present at 500°F, and intercrystalline cracking takes place in a narrow temperature-strain rate range (near 500°F), but failure is generally by transgranular means. At 700°F and above, intercrystalline cracking disappears, but extensive void formation is still evident.<sup>19</sup>

(c) Higher additions of alloy (copper, magnesium, zinc) in solid solution sharply increase the amount of intercrystalline cracking at 500°F and cause intercrystalline failure over a narrow temperature-strain rate range. Voids are small and numerous in these higher alloy materials but have been less extensively investigated thus far.

There is neither macroscopic nor microscopic evidence to indicate less perfect boundaries in the higher alloyed materials; they appear equally plane and regular.

Grain-boundary roughening, or corrugation, has been observed for quite some time.  $^{9,20,21}$  The extent to which it occurs shows up vividly in Fig. 7 for an aluminum-5% magnesium alloy, which was strained

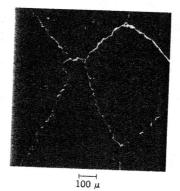


Fig. 7. Corrugated grain boundaries in aluminum-5.1% magnesium alloy after 10.8% elongation at 546°F. Dark-field photomicrograph.

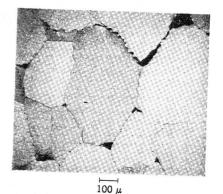


Fig. 8. Aluminum-20% zinc alloy tested in creep rupture at 500°F. Rupture life, 12 hr. Stress direction is vertical.

10.8% in creep at 546°F and then photographed with dark-field illumination. Figure 8 shows that intercrystalline cracks are often associated with such corrugations. These cracks, as shown in Fig. 8, appear to be on one side only of the grain boundary for an aluminum-20% zinc alloy, at such a position on each corrugation that the long axis of the crack is nearly normal to the stress direction. 23

Observations by Brunner <sup>22</sup> and Mullendore <sup>19</sup> of a 2% magnesium-aluminum alloy clearly show the following:

(a) Corrugations occur gradually along a particular grain boundary, increasing in number but maintaining about the same wavelength. These corrugations have been observed to be equivalent in size either to the slip spacing <sup>22</sup> or to the subgrain size. <sup>20,22,24</sup> It is possible that the latter condition prevails in the absence of coarse slip <sup>22</sup> (Figs. 9 and 10).

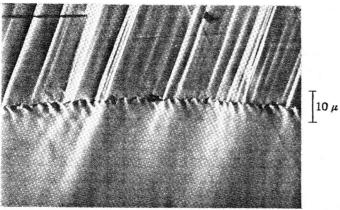


Fig. 9. Grain-boundary corrugations caused by creep at  $400^{\circ}\text{F}$  and 6800 psi in aluminum-1.9% magnesium alloy. Preliminary creep clongation, 9.4%, followed by repolishing and 10.2% additional elongation. Oblique illumination.

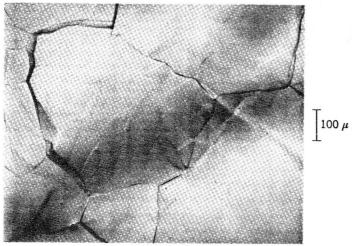


Fig. 10. High-purity aluminum repolished after preliminary creep elongation of 9.8% at  $700\,^{\circ}\text{F}$  and 250 psi, then further elongated 1.6% in creep. Oblique illumination.

(b) Voids are first noted only after significant creep strain (of the order of 5%) and after the corrugations are well developed.

(c) The voids are of fairly uniform size (one or two sizes), quite spherical, and are all in one grain only <sup>19</sup> (Fig. 11). They are located at points comparable to those of the cracks in Fig. 8.

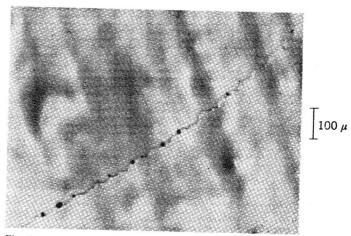


Fig. 11. Aluminum-2% magnesium alloy tested in creep rupture at 500°F and 4000 psi. Rupture life, 5 hr. Stress direction is vertical.

(d) Depending on the conditions of stress and temperature, voids may be entirely isolated from one another, joined by a grain-boundary crack, or so closely spaced that linking may be construed but not proved.

#### Discussion

Voids and intercrystalline cracks are not uniquely associated. Transgranular failures and the complete absence of intercrystalline cracks are observable under test conditions that can produce numerous voids.

It is not clear whether slip is always a prerequisite for the formation of a jog to initiate the void mechanism, as proposed by Gifkins. It appears that considerable creep must take place (5% in the above instance) before voids are observed and that grain-boundary sliding is a definite prior condition. However, grain-boundary sliding and granular slip are not sufficient to assure void formation. If grain-boundary migration takes place at a rate greater than some minimum value, voids are not formed (nor are grain-boundary cracks started or propagated).

In the absence of gross natural restraints to boundary sliding and migration by such things as triple points, secondary phases, and irregular grain boundaries, the imperfect nature of the grain boundary (on an atomic scale) can conceivably provide the means for initiating grain-boundary corrugations, which then serve as jogs leading to dislocation pile-ups and crack formation. These corrugations can be initiated either by slip or by subgrain boundaries.

In line with Machlin's calculations,16 there is reason to believe that

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voids do not start by vacancy condensation. Instead, actual cracks are initiated by atomic parting under conditions of dislocation pile-up to a point where the cohesive stress of the boundary is exceeded, in a manner similar to that indicated for granular fracture by Gilman.25 At high temperatures, if further dislocation pile-up does not take place, the crack coalesces into a spherical void by surface tension or vacancy condensation.15 If conditions of dislocation pile-up continue, the crack grows as a crack. Boundary migration can annul the effects by isolating the

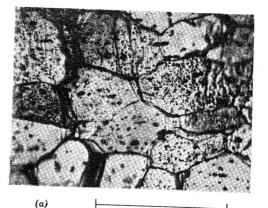
Because of the cyclic nature of grain-boundary sliding and migration, 28 crack propagation or growth in ductile materials is also cyclic. This is supported by the appearance of the fractured specimens, as shown in Fig. 3, wherein a multitude of fine cracks throughout the specimen can be seen, most of them of rather uniform size, at some stage prior to actual fracture. The actual observation of crack propagation in aluminum alloys under creep conditions also shows this cyclic process of crack propagation.27

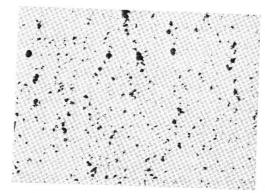
There is abundant micrographic evidence of the fact that the shape of an intercrystalline crack is, to a marked extent, the combined effect of temperature, time, strain rate, and strain. A longitudinal section in a ductile metal (2S-O aluminum), which is subject to intercrystalline cracking, is noted in Fig. 12, where view (a), which is well away from the fracture, shows typical sharp, intercrystalline cracks normal to the stress direction (stress axis is horizontal). View (b) shows rounded cracks ("voids") nearer to the point of fracture; the cracks have grown to this shape by surface tension, and/or vacancy migration, and strain. View (c) shows the further effect of strain on the original sharp grainboundary microcrack very near the fracture. The cracks are now elongated in the direction of the axis of tension.

The appearance of rounded voids "during creep" may better be construed as evidence of a form of recovery, or at least as a decrease of stress concentration of a prior sharp intercrystalline crack. It is unlikely that such voids would link by lateral growth into finite cracks or lead to failure unless the cross section of the specimen is drastically changed.

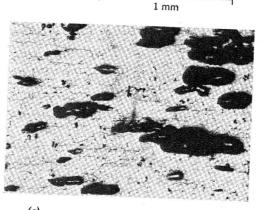
It is proposed that the fracture mechanism calls for a basic material condition such as one in which the grain is a weaker entity than the grain boundary at low temperatures and stronger at high temperatures. The relative orientations of any two grains forming a boundary determine the degree of imperfection or misfit existing, thereby establishing an unequal strength condition with respect to both a shear and a normal

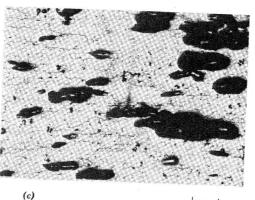
Grain-boundary sliding occurs either in small steps that are established





(b)





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Fig. 12. Appearance of cracks and voids in 2S-O aluminum, tested at 910°F and 500 psi. (a) Sharp cracks well removed from the fracture interface. (b) Rounded "voids" much nearer the fracture interface. (c) Elongated "voids" very near the fracture interface.

by jogs in the grain boundary, or between blocking points such as inclusions or other excess phases, or by wholesale shear along the entire length of a straight grain boundary. In the latter case, there is a larger stress concentration at the triple points. Slip also causes a pile-up of dislocations on the boundary and causes a stress concentration on the grain boundary. These stresses cause one grain to deform more, and grainboundary migration into this grain takes place. If the migration is rapid and on a broad front, the intercrystalline cracks do not form. If the migration process is slower, or local, then corrugations are formed, these being potential sources of cracks at the apexes or at other points where the stress is nearly normal to the boundary.6 Cracking proceeds slowly in this instance, and, by linking of normal and shear stress regions, gives the uniform cell-type fracture discussed by Chang and Grant.<sup>28</sup> If migration is largely restricted or virtually absent, this condition being more typical of complex commercial materials, grain-boundary sliding takes place in very small increments, with the pile-up of dislocations resulting in high stresses either from slip or boundary shear. The stiffness of grain-boundary junctures gives rise to very high stresses at that point, resulting in intergranular fracture. Yet, even in this instance, the first such crack does not run to failure but advances at a finite rate in proportion to the ductility of the material. As a result, there is time for the dissipation of dislocation pile-up either by climb, by thermal recovery (including surface tension effects), by initiation of slip, or possibly, by vacancy condensation that blunts the tip of the crack. In this way, a material with only 5 to 10% elongation at fracture still shows a multitude of intercrystalline cracks instead of one or only a few intercrystalline cracks that run to failure.

If this mechanism is approximately correct, any of the more recent quantitative approaches for calculating the stress induced by dislocation pile-up might serve. The recent review of intergranular fracture processes in creep by Davies and Dennison <sup>29</sup> would particularly apply.

#### **ACKNOWLEDGMENTS**

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#### DISCUSSION

E. E. Underwood, *Battelle Memorial Institute*. There is a remarkable similarity between Grant's figure showing a "scalloped" grain boundary with spherical voids at the cusps, and the well-known picture of a "scalloped" grain boundary with precipitate particles located at the cusps.

The spherical voids may have developed from the precipitate particles that were originally retarding the motion of the grain boundary.

Particle dissolution could occur by the "solute atom-vacancy" interchange mechanism,1 which would be promoted by the proximity of a vacancy source (the grain boundary) and the stress-induced diffusion. Contrary to the implication made by Grant, we are not dealing here with the larger particles. The particles that are effective in retarding grainboundary migration are the small ones, and these small ones are the first to dissolve when particle growth and coalescence occur.

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