Communications

Origin of Brittle Intergranular Fatigue Fracture in Warm Aged Al-3.6 Wt Pct Cu

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When a binary Al-3.6 wt pct Cu alloy is aged for a sufficiently long time at 130 to 190°C, the failure mode is intergranular during monotonic tensile testing. Intergranular failure during fatigue crack propagation has also been reported elsewhere.[1-3] In specimens aged for various times at 160 and 190°C, propagation of fatigue cracks occurs intergranularly provided ΔK is sufficiently large.[4] Typically, in pull-pull specimens the failure mode at low ΔK is transgranular, becomes mixed as ΔK is increased, and is completely intergranular at high ΔK. The transition range of ΔK decreases as the aging time is increased, as shown in Table I for 190°C aging, and for a given aging time the transition range of ΔK decreases as the aging temperature is increased.

Past investigators[5-7] have attributed the metallurgical cause for this mode of fracture to either grain boundary precipitation or a precipitate free zone (PFZ). In the present investigation, high magnification SEM fractography studies of grain boundary fracture surfaces were compared with TEM results, for areas including grain boundaries, to identify the source of grain boundary embrittlement in samples aged for various times at 160 and 190°C.

To examine the grain boundaries of fatigue crack propagation specimens, sections 0.5 mm thick were cut parallel to and 1.5 to 3 mm below the fracture plane using electrical discharge machining. The resulting 0.5 × 3 × 10 mm slabs were subsequently electropolished to a thickness of 0.13 mm to remove the spark damaged region. A modified Bollmann technique was then used to selectively thin an area in the center of the 3 mm wide slab. The electrolyte was a mixture of 20 vol pct perchloric acid (70 pct absolute) and 80 vol pct ethanol cooled to below 0°C. Immersion in an electrolyte of 25 vol pct nitric acid-75 vol pct methanol cooled to below 0°C at a potential of 8 VDC was found effective in removing any surface film which formed during thinning.

In samples aged for two days at 160°C, when ΔK was 10 MN/m$^{3/2}$, approximately a third of the fracture surface exhibited the transgranular fracture mode. On aging seven days at 160°C, however, the transition to 100 pct intergranular fracture was complete at 10 MN/m$^{3/2}$. In both cases TEM tilting experiments showed that there was no appreciable PFZ. Fig. 1 gives the results for three different tilt angles of a particular grain boundary area in a specimen aged at 160°C for two days. Examination of all three micrographs shows that no PFZ is actually present. The figure also clearly shows particles in the grain

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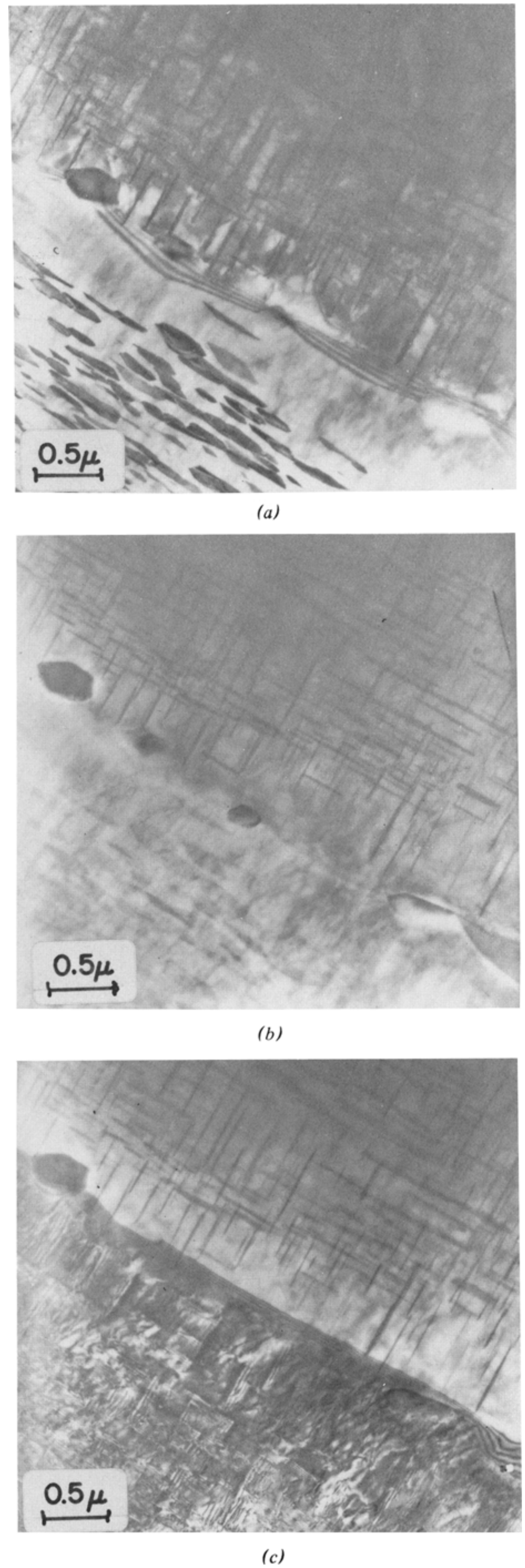


Fig. 1—TEM micrographs of typical grain boundary area for Al-3.6 wt pct Cu aged at 160°C for 48 h with a magnification of 20,000 times: (a) 0 deg tilt, (b) 1 deg 40 min tilt, and (c) 4 deg 10 min tilt.

Table I. Fracture Mode as a Function of ΔK and da/dN for Al-3.6 Wt Pct Cu Aged at 190°C

	Aging Time, 12 h		Aging Time, 36 h		Aging Time, 54 h	
Pct Intergranular Fracture	ΔK	da/dN	ΔK	da/dN	ΔK	da/dN
0	6 to 10	0.2 to 1.6	4 to 10	1.0 to 4.6	4 to 9	0.2 to 2.0
33	10 to 14	1.6 to 6.7	10 to 14	4.6 to 14		
50	14 to 17	6.7 to 30	14 to 22	14 to 120	9 to 11	2.0 to 5.6
67	17 to 34	30 to ∞				
100	none	none	22 to 33	120 to ∞	11 to 28	5.6 to ∞

ΔK values are given in units of $MN/m^{3/2}$, values of crack propagation rates da/dN are in units of 10^{-2} μm/cycle.

Table II. Mechanical and Microstructural Properties of Two Al-3.6 Wt Pct Cu Alloys

Heat Treatment	s, Particle Spacing, μm	h, Particle Size, μm	σ_u MPa	K_c MPa$\sqrt{m}$	$f_a^{-1/2} \propto \frac{s+h}{h}$	$f_a^{1/2}\,\sigma_u$ (MPa)	K_c^2 ($MPa^2\,m$)
Two days, 160°C	0.5	0.3	299	33	2.66	795	1089
36 h, 190°C	0.5	0.5	289	30	2.00	578	900

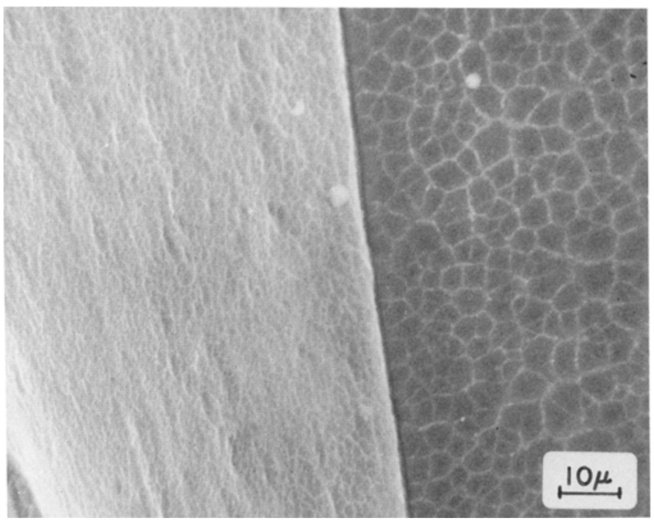


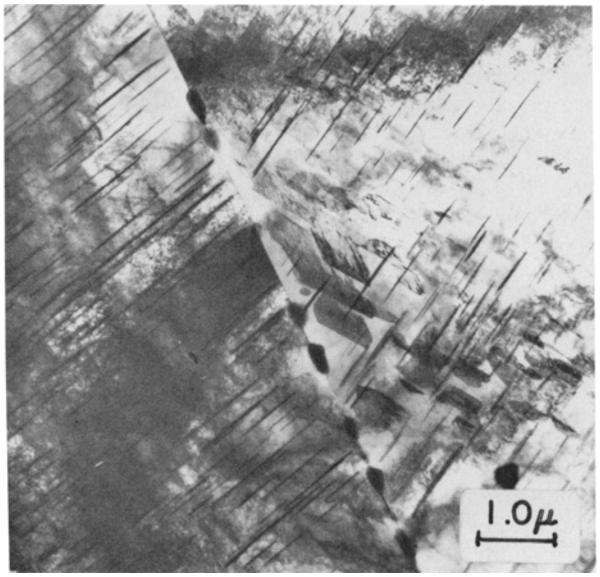
Fig. 2—SEM fractograph of grain boundary failure in Al-3.6 wt pct Cu aged 48 h at 160°C, magnification 780 times. This structure is identical for all areas exhibiting this fracture mode.

boundaries approximately 0.3 μm in diam and spaced approximately 0.5 μm apart.

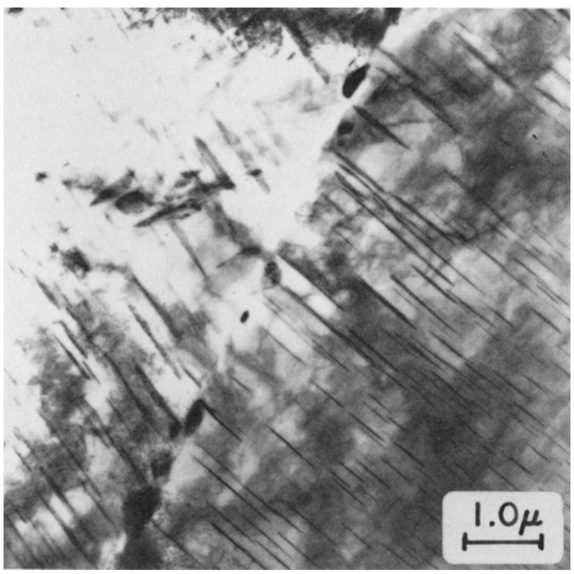
Fig. 2 shows a typical high magnification SEM fractograph of the brittle intergranular fracture surface found in a fatigue crack propagation specimen aged two days at 160°C. The two adjacent planar surfaces of the grains are approximately 45 deg with respect to the plane of the photograph. Networks of ridges outlining pitted areas are observed on both sides of the boundary. These appear as equiaxed tensile dimples forming a cell-like structure of 5 μm diam on the right-hand side and 0.5 μm diam on the left-hand side. Presumably, the voids associated with the dimple formation initiate at the precipitates in the grain boundary. The grain boundary precipitates shown in the TEM micrographs are approximately 0.5 μm apart which is smaller than the diameter of the cell-like structure on the right-hand side; however, the grain on the left-hand side of the boundary shows the periodicity predicted for ductile void coalescence is actually present. The correct tilting angle seems to be needed to see the finer structure. Thus, the grain boundary embrittlement is clearly identified with the precipitate phase found in the grain boundary and cannot be attributed to a PFZ since this feature was not observed on aging at 160°C.

Study of specimens aged at 190°C leads to a similar conclusion. Fatigue crack propagation specimens aged for either 12, 36, or 54 h at 190°C showed partial brittle intergranular fracture at the lowest stress intensities tested, 5 $MN/m^{3/2}$ (Ref. 4). The grain boundary microstructures observed in all three were similar to that found in the specimens aged at 160°C. Figs. 3(a) and (b) show TEM micrographs of a grain boundary area with two different tilt angles for aging 36 h at 190°C. Again, there is no clearly defined PFZ. The grain boundary particles are 0.5 μm in diam which is slightly larger than the 0.3 μm diameter grain boundary particles formed during the 160°C aging; but the spacing is approximately the same, namely, 0.5 μm. A high magnification SEM fractograph of an intergranularly failed region, Fig. 4, shows a similar topography to that shown in Fig. 2. The grain boundary embrittlement on aging at 190°C, as at 160°C, is due to the precipitate structure at the grain boundary. The absence of a PFZ when θ' is precipitated on aging at 160 and 190°C is in agreement with the data reviewed by Kelly and Nicholson[8] and others.[9-12] The θ' precipitate is initially distributed preferentially in regions adjacent to grain boundaries but at peak hardness this heterogeneity is less apparent due to high density of precipitates inside the grains. Both Figs. 1 and 3 essentially show a homogeneous distribution of θ' up to the grain boundaries.

The idea that the brittleness of Al-Cu binary alloys is caused by the presence of compounds at the grain boundaries is not new.[13] However, what is new is that the size of these grain boundary particles need not be large. The fractographic evidence shows the grain boundary fails by voids initiated at particles on the order of tenths of microns in size, as observed in other cases of ductile fracture.[14] A necessary condition for ductile grain boundary failure is that voids form more easily around particles in the grain



(a)



(b)

Fig. 3—TEM micrographs of typical grain boundary area for Al-3.6 wt pct Cu aged at 190°C for 36 h with a magnification of 10,000 times: (*a*) 5 deg tilt and (*b*) 10 deg tilt.

boundary than around particles in the crystal. An advancing fatigue crack gives rise to an increasingly large amount of displacement at the crack tip (*i.e.*, crack tip opening displacement) which must be accommodated by strain or fracture. When nondeforming particles are present, they give rise to larger stress concentrations which are less easily accommodated by dislocation motion in the grain boundary than in the grain itself. Thus the increase in the percent grain boundary fracture with increase in ΔK is not surprising.

Embury and Nes[15] published a simple model for grain boundary fracture based on the nucleation and growth of voids at grain boundary precipitates. The essential feature of their approach predicts that the square of K_c should be proportional to the product of the ultimate strength times the inverse square root

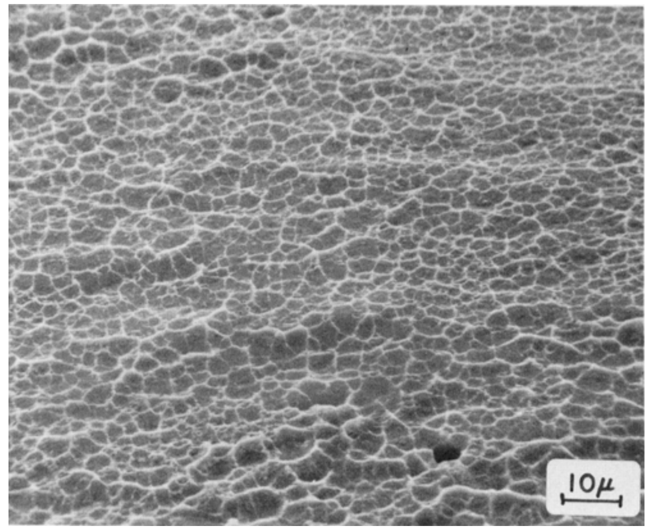


Fig. 4—SEM fractograph of grain boundary failure in Al-3.6 wt pct Cu aged 54 h at 190°C with a magnification of 780 times. This structure is identical for all areas exhibiting this fracture mode.

of the area fraction, f_a, of grain boundary precipitates. Following Embury and Nes, the area fraction can be related to the grain boundary particle spacing, s, and size, h. The last two columns of Table II compare K_c^2 and $\sigma_u f_a^{-1/2}$ for two days at 160°C and 36 h at 190°C. The ratios of K_c^2 are within 12 pct of the ratios of $\sigma_u f_a^{-1/2}$ for both treatments. Since the data is limited to only two heat treatments, these results cannot be taken as confirming Embury and Nes; however, the results are encouraging.

Calabrese and Laird[3] suggested that crack initiation in grain boundaries of binary Al base-Cu alloys with θ' precipitates formed by aging at 240°C is due to intense slip at precipitate free areas near the grain boundaries. However, they did not document the presence of PFZ. In view of the present results, it seems more probable that the presence of precipitates in the grain boundaries like those shown in Figs. 1 and 3 serve as stress concentrators and are responsible for the early crack initiation which they observed.

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Positron Lifetime Studies Made in Fatigue Damaged AISI 4340 Samples

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In the last few years a number of positron studies have been performed to observe defects in metals. These studies have been limited primarily to the examination of point and/or line defects for which excellent reviews exist.[1,2] Only a few articles have appeared in which the positron technique has been employed to study other more complicated defect structures.[3-6]

It has been suggested previously[7] that positron studies could eventually lead to a new nondestructive testing technique. It is now recognized, however, that such studies can only be useful in stages of deformation prior to the occurrence of the saturation of positron trapping at dislocations.[8] This saturation limit seems to be determined by many factors including grain size,[9] deformation process, and most probably other variables such as the type of substructure. Clearly, the situation is not presently well understood.

In this study the primary positron lifetime in AISI 4340 steel, in each of hardness levels Rockwell *C* (Rc) 27 and 51, was measured as a function of number of cantilever bending fatigue cycles with a maximum stress of two-thirds of the yield stress for each condition. In a previous study,[4] of annealed Ni and a Ni-Co alloy, the positron lifetime increased quickly and saturated at or before 7 pct of the total fatigue life, when cycled in the cantilever mode with maximum stresses equal to or greater than the yield stress.

With this previous result in mind we examined the Rc-27 condition to observe if saturation of the positron lifetime occurred for a maximum cyclic stress level below the yield stress. The Rc-51 sample was selected to determine if a decrease in the positron lifetime would occur. Such a decrease in positron lifetime during fatigue cycling would support the earlier report of Klesnil and Lukas[10] who found cyclic fatigue softening in hardened low carbon steel. This softening was accompanied by the sharpening of Debye X-ray diffraction rings. Such sharpening is commonly associated with an increase in X-ray particle size with concomittant decrease in dislocation density.[11-13] Other workers[14,15] have associated fatigue softening with decreasing dislocation density and decreasing complexity of dislocation substructure determined from direct transmission electron microscope observations.

The fatigue samples were cut from the same ingot and machined into constant maximum stress bending fatigue specimens. The samples were then austenitized at 1116 K for 1 h. The Rc-27 sample was subsequently cooled in lime and then tempered at 922 K for 1 h. The Rc-51 sample was oil quenched and tempered for 1 hour at 682 K. From the hardness measurements the yield strength levels were estimated to be approximately 1526 and 869 MN/m^2 for the Rc-51 and Rc-27 samples respectively. The Rc-27 samples were immersed in a 10 pct solution of H_2SO_4 in distilled water at 353 K for 5 min to remove some surface mill-scale. Both sets of samples were polished with 00 emery paper after heat treatment to remove surface imperfections, and then cleaned thoroughly with acetone to remove surface grease and fine particulate matter.

A Sontag SF-2-U fatigue machine with a fixed frequency of 30 Hz was employed for cycling. The sample dimensions are described elsewhere.[16] After each fatigue run the samples were carefully removed from the fatigue machine and introduced into the positron lifetime system in the standard sandwich configuration.[4] The $Na^{22}Cl$ source was enclosed in 0.005 in. (0.013 cm) mylar sheets and then inserted between the fatigue specimens. Each measurement lasted approximately 16 h.

The constant fraction discriminator designed by Hall[17] gave very reproducible results and ultimately allowed the run time to be reduced by a factor of three. The times between the positron measurements were not always constant probably causing some error which will be described later.

The data were analyzed by a computer program which is a modification of "PositronFit" by Kirkegaard and Eldrup[18] and is described elsewhere.[16] The data were corrected for a linear background and annihilation times appropriate for both annihilations in the mylar and in the $Na^{22}Cl$ positron source.

As expected, fatigue hardening of the soft Rc-27 samples caused the positron lifetime to increase as seen in Fig. 1. The initial lifetime was approximately 119 ps and at fracture was 165 ps, the change indicating an increase in the number of defects. The decrease in slope of the curve in Fig. 1 occurs at about 20 pct of the total fatigue life. In earlier work[4] in the plastic range the slope change occurred earlier (~7 pct of total fatigue life) and was complete, *i.e.*, the slope went to zero, indicating saturation. Fig. 1 shows that during elastic fatigue the slope decrease is not so drastic. In fact the small positive slope during the last 80 pct of fatigue life promises to be a very useful quality for failure prediction and design purposes.

With the Rc-51 specimens we hoped to study fatigue softening. The initial mean positron lifetime of 205 ps before fatiguing was probably due to the fact that a complicated and intense defect structure is introduced during the oil quench. Fig. 2 shows a decrease of approximately 37 ps in the mean positron lifetime

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