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An experimental investigation of the orthogonal (diamond) grain configuration in high temperature fatigue

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Experiments on polycrystalline aluminium show that the cyclic grain boundary migration observed in high temperature fatigue leads ultimately to the development of the orthogonal (diamond) grain configuration. Angular measurements demonstrate that the grain boundaries migrate so that a large proportion (~ 25%) lies in the angular range of 40 to 50° to the stress axis. Grain growth may occur during the migration process by the elimination of some small grains, although other small grains may increase in size when migration occurs in an outwards direction. Migration is initially rapid, but the rate of migration decreases after large numbers of fatigue cycles. As the rate of migration slows down, the grains become divided into subgrains, and the subgrain boundaries tend also to exhibit an orthogonal configuration.

1. Introduction

It is now well established that grain boundary migration is a very important deformation process in metals subjected to fatigue at elevated temperatures. Two different and distinct types of experimental observation have been reported to date.

First, it was demonstrated in early experiments on aluminium by Ritter and Grant [1] that the migration is cyclic in nature, and that there is an approximate one-to-one correspondence between the number of migration markings visible at any selected grain boundary on the specimen surface and the number of fatigue cycles imposed on the specimen. This effect was subsequently confirmed by Wigmore and Smith [2] using OFHC copper, and there have been several recent investigations of the cyclic behaviour using aluminium [3, 4], Mg–0.8% Al [15] and high purity lead [5–8]. A detailed microstructural investigation of aluminium has shown that the number of sharp migration markings occurring at each grain boundary is equal to $(N + 1 + M)$, where N is the number of loading cycles and M is the number of migration reversals during the test [9]. In practice, M is usually equal to zero so that there are generally $(N + 1)$ sharp markings after N loading cycles.

Second, it was reported in very early experiments on lead in high temperature fatigue that the grain boundaries tend to migrate from a random distribution to take up orientations on the surface which are close to 45° to the stress axis [10]. This distribution is generally known as the orthogonal or diamond grain configuration, and it has been documented in a wide range of metals: aluminium [1, 11–14], cadmium [15], copper and copper alloys [2, 14, 16–21], α-Fe [22–24], Mg–0.8% Al [16, 25, 26], nickel [27], lead [11, 12, 21, 28–34], stainless steel [35, 36], zinc [37], Zn–0.1% Cu [14], zirconium [20, 38, 39] and Zircaloy-2 [39].

An important distinction between these two different sets of observations of migration in high temperature fatigue lies in the total number of testing cycles: whereas reports of cyclic migration markings are generally restricted to observations recorded at <100 fatigue cycles and the maximum deformation examined to date is 150 cycles in

*Present address: Department of Materials Science and Engineering, School of Engineering, Shiraz University, Shiraz, Iran tests on aluminium [4], the observations of the orthogonal grain configuation usually relate to conditions at fracture or at least at $> 10^4$ cycles.† Furthermore, with only two exceptions [17, 22], the various angular distributions which have been presented to support the occurrence of the orthogonal configuration were obtained at $> 10^4$ cycles [15, 16, 19, 21, 37]. The two exceptions are the angular measurements on copper and a copper alloy by Cocks and Taplin [17] and on α-Fe by Westwood and Taplin [22], but these two investigations were not generally representative because, in all three materials, fracture occurred after a total of only ~ 100 to 200 fatigue cycles. Thus, for those materials exhibiting a fatigue life of $> 10^3$ to 10^4 cycles, there is no clear link between the observations of cyclic migration in the very early stages of fatigue testing and the reports of an orthogonal grain configuration occurring at failure or, at best, after $\sim 10^4$ cycles.

The present investigation was conducted to provide a direct link between these two sets of observations. Specifically, the investigation was carried out with two objectives: (i) to examine the development of the orthogonal configuration in high temperature fatigue by taking a series of photomicrographs of the same areas of the specimen over a very wide range of loading cycles and (ii) to determine the change in the angular distribution of the grain boundaries with respect to the stress axis by taking measurements from the pretest condition up to $> 10^3$ cycles.

2. Experimental material and procedure

The experiments were conducted using aluminium of 99.99% purity. The material was prepared from a production ingot, ~ 20 cm thick, by hot rolling at ~ 700 K to a thickness of ~ 3.0 mm, air cooling, and cold rolling to a final thickness of 1.8 mm. The following impurities were revealed in the final material by a semi-quantitative spectrographic analysis (in ppm): Cu 30, Fe < 10, Mg 20, Mn < 10, Si 30 and Zn 40.

Test specimens were cut from the sheet to the profile and dimensions given in Fig. 1, and the tests were performed using a simple reverse bending machine described in detail elsewhere [40]. Briefly, the specimen was held rigidly in the testing machine by the larger end, and the smaller end was attached to a moving plate so that it was forced to move in a backwards and forwards motion. The specimen profile was designed so that, when the specimen is bent into an arc of a circle which is perpendicular to the section shown in Fig. 1, the strain is uniform over the short test section shown shaded. All of the experimental observations were recorded within this test section.

The testing machine was operated through a

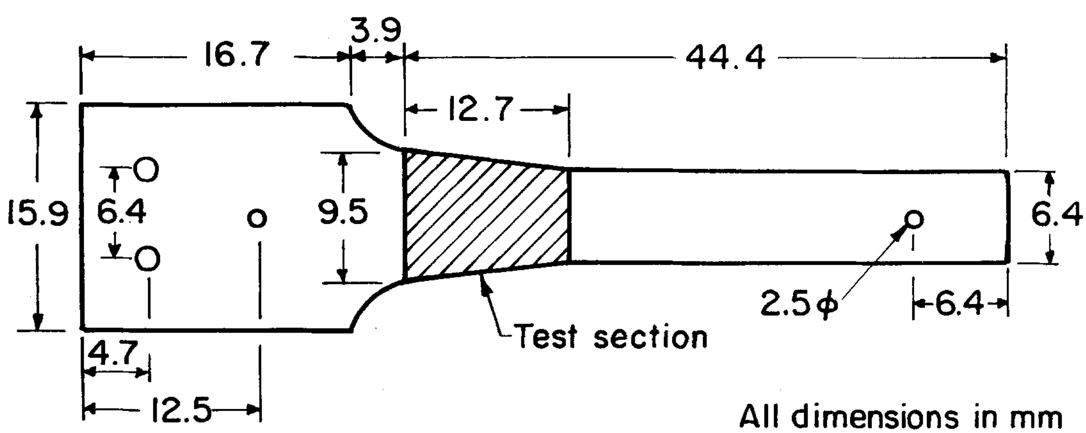


Figure 1 Specimen profile and dimensions: all of the experimental observations were recorded within the test section where the strain is uniform.

†In this report, the term "orthogonal grain configuration" is preferred to the more usual "diamond grain configuration" because of the observation by Singh *et al.* [14] that the "diamond" grains are not four-sided nor is there a preponderance of four-fold junctions. On the contrary, it was shown that the configuration represents a distortion of the usual annealed condition so that, on a longitudinal section, there is a large percentage of the total lengths of the grain boundaries lying near to 45° to the stress axis.

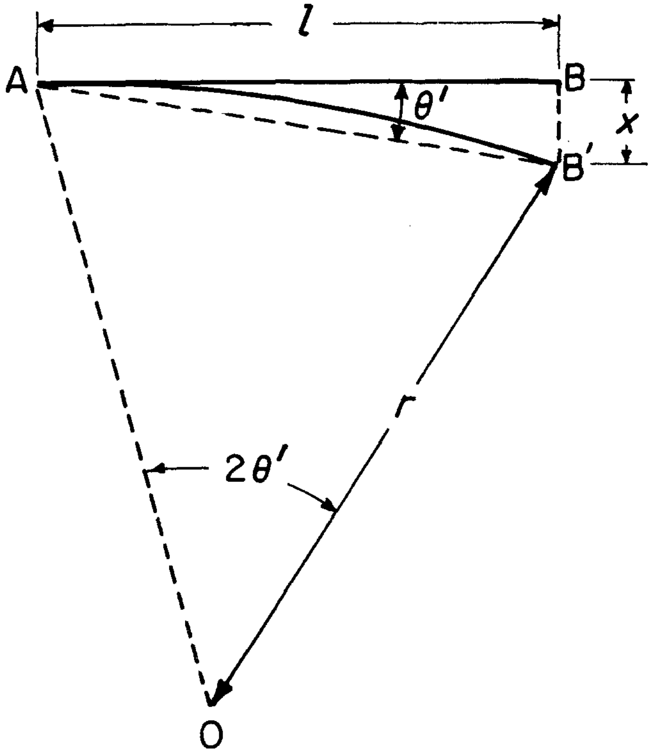


Figure 2 Method used to estimate the strain amplitude, $\Delta\epsilon$: the test section AB is bent into an arc of a circle AB′ centred at 0.

small electric motor, controlled by a calibrated speed reducer, which was used to rotate an eccentric disc connected rigidly to the plate at the smaller end of the specimen. As the specimen was subjected to a series of cycles of reverse bending, the total number of cycles occurring during the test was recorded continuously using a mechanical counter attached to the eccentric disc.

Fig. 2 illustrates schematically the method of calculating the strain amplitude. When the short test section, AB, is bent into an arc of a circle, AB′ centred at 0, due to a deflection of x at the smaller end, the strain is given by [41]

$$\Delta\epsilon = \frac{\zeta}{2r} \tag{1}$$

where ζ is the specimen thickness (1.8 mm) and r is the radius of curvature. The value of r may be approximated as

$$r \simeq \frac{360}{2\theta'} l \tag{2}$$

where θ' is the deflection angle ($= \tan^{-1} x/l$) and l is the length of the test section (12.7 mm). Equation 2 is an acceptable approximation provided x is small; in the present work, the strain amplitude was set at $\Delta\epsilon = \pm 0.25\%$.

Prior to testing, the specimens were annealed at 723 K for 24 h to give a mean linear intercept grain size, d, of 200 μm. The specimens were carefully electropolished before testing using a solution of 20% perchloric acid in methanol, a stainless steel cathode, and with the temperature of the electrolyte maintained at $-80°$ C using a bath of dry ice and acetone.

The tests were performed in air using a vertical single-zone furnace placed around the specimen to give a testing temperature of 573 ± 2 K. For each test, the specimen was held at temperature for 30 min before cyclic loading to establish thermal equilibrium, and the temperature was continuously monitored by placing a thermocouple adjacent to the test section.

Two different types of observation were carried out using two different specimens.

Initially, the testing procedure consisted of bending a specimen through a selected number of whole cycles at a frequency, f, of 1.67×10^{-2} Hz, removing the specimen from the testing machine, and taking a series of photomicrographs at various points within the test section. The specimen was returned to the machine, testing was continued, and the test was then interrupted again and photomicrographs were taken at the same points in the test section. This process was continued up to $> 10^3$ cycles and it should be noted that the specimen was always examined at zero strain amplitude corresponding to a whole number of loading cycles.

Using this procedure, it was found that the specimen surface tended to become fairly corrugated after large numbers of testing cycles and it was then necessary to remove a thin surface level by lightly abrading with 0.05 μm alumina powder followed by a very short electropolish. The depth removed by this procedure was very small and, as will be seen from the photomicrographs, this light polish had no apparent effect on the subsequent microstructural observations.

The second set of observations was designed to check the rate of migration of the grain boundaries into the orthogonal grain configuration. A specimen was electropolished and etched, and measurements were taken to determine the angular distribution of the boundaries on the specimen surface with respect to the longitudinal axis. The etching was carried out using a solution of 2% HF, 25% HNO_3 and 73% H_2O by volume, and measurements were made along a series of random traverses by recording the various angles, θ, between the traces of the grain boundaries on the polished surface and the longitudinal axis of the specimen. Similar measurements were taken after testing at a frequency of 3.30×10^{-2} Hz through 100, 800 and 2500 cycles, respectively, and at each point the specimen was polished and lightly etched to reveal the boundary configuration. In each condition, individual measurements were recorded at a total of more than 300 separate boundaries, and the results were plotted as histograms by dividing the readings into angular increments of 10°.

3. Experimental results

3.1. Metallographic observations

To obtain information on the role of cyclic migration in moving the grain boundaries into the orthogonal grain configuration, a series of photomicrographs was taken of the same areas within the test section for a testing sequence from an initial 3 cycles to a total of 1132 cycles: an example of a complete sequence is shown in Fig. 3, where each photomicrograph shows the same area at the same magnification.

The specimen was tested at 573 K with a strain amplitude of ± 0.25% and a frequency of 1.7×10^{-2} Hz. The first photomicrograph, shown in Fig. 3a, was taken after 3 cycles of deformation, and this serves to show the initial grain configuration in the pre-test condition. It is clear that very

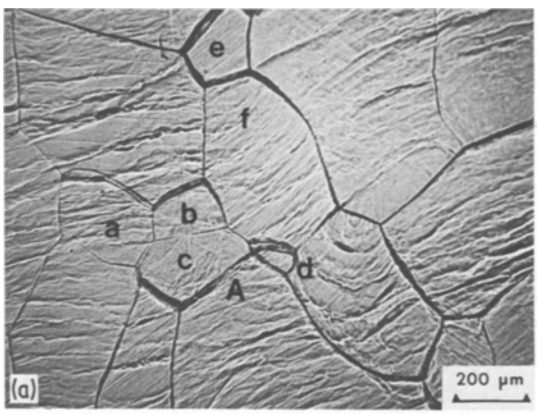
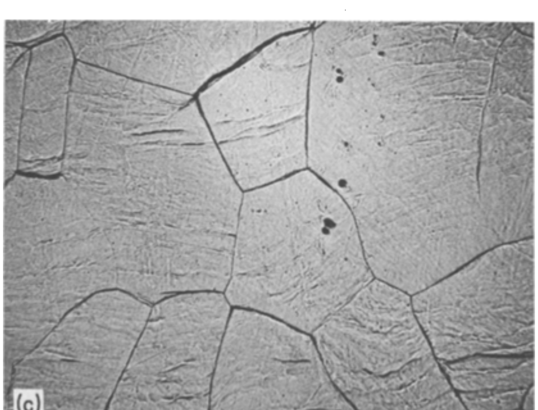


Figure 3 The change in grain configuration in aluminium due to testing at 573 K with a strain amplitude of ± 0.25% and a frequency of 1.7×10^{-2} Hz: the same area is shown after (a) 3 cycles, (b) 73 cycles, (c) 76 cycles (with a light polish after 73 cycles), (d) 126 cycles, (e) 426 cycles, (f) 429 cycles (with a light polish after 426 cycles), (g) 729 cycles, (h) 732 cycles (with a light polish after 729 cycles) and (i) 1132 cycles.



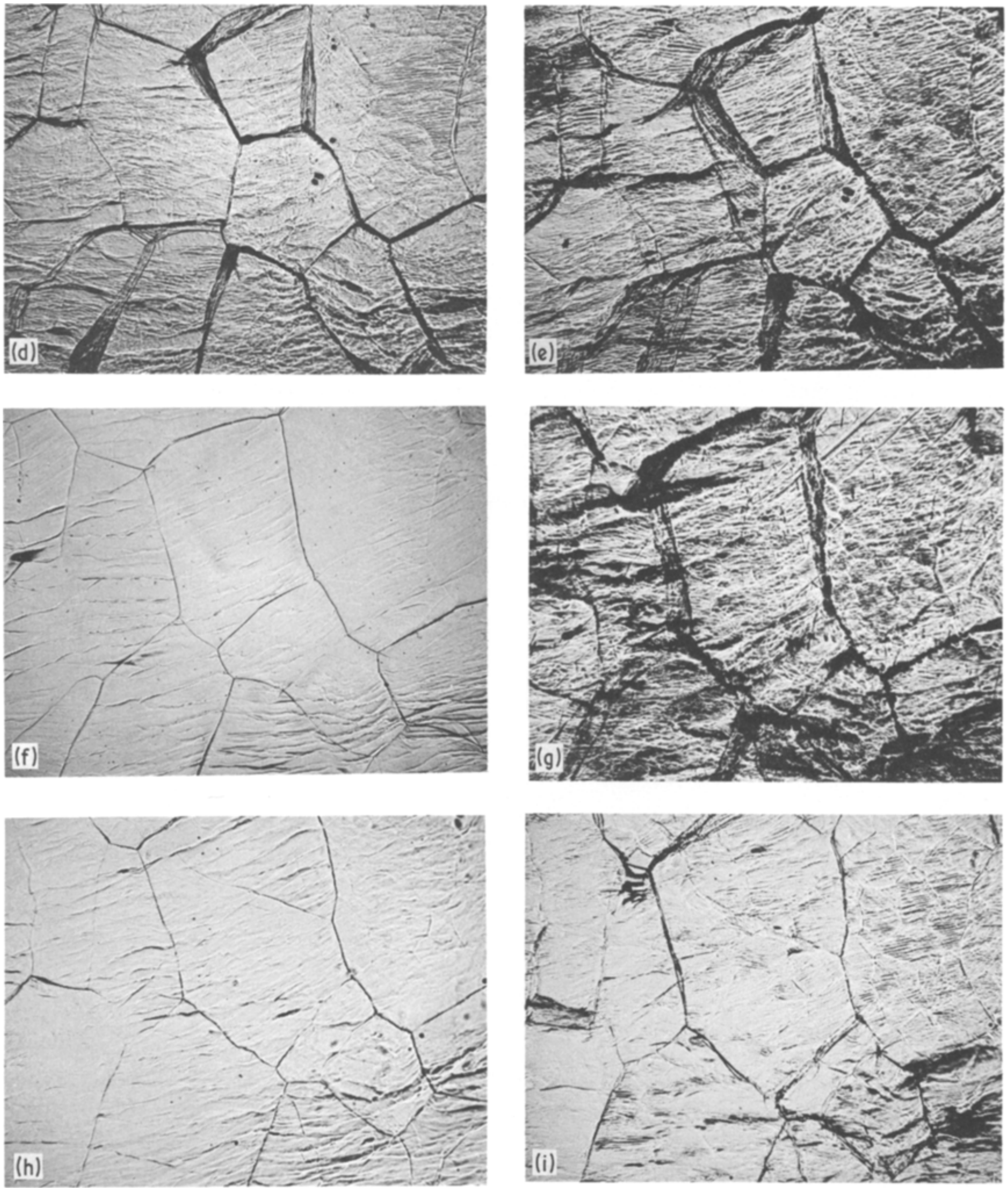


Figure 3 Continued.

extensive slip occurs within the grains in the initial 3 cycles, and there is also extensive migration at several of the grain boundaries. Inspection at a higher magnification showed that, as noted previously [9], there are $(N+1)=4$ sharp migration markings at each of the migrating boundaries, where N is the number of loading cycles: an example of this effect is visible at the grain boundary labelled A.

The occurrence of extensive grain boundary migration leads to grain growth in fatigue, as noted in earlier studies [3, 9]. Examples of the grain growth process may be noted by reference to the four smaller grains labelled a, b, c and d in Fig. 3a which, as will be demonstrated, quickly disappear by the migration of boundaries across the widths of the grains. On the other hand, it will be shown also that some small grains may increase in size and grow at the expense of their larger neighbours. An example of this effect is given by the two grains labelled e and f in Fig. 3a, since it will be demonstrated that the smaller grain e grows at the expense both of the larger grain f and of the other large surrounding grains.

Fig. 3b shows the same field of view after 73 loading cycles. There is extensive slip, and also very extensive boundary migration especially in the vicinities of grains a to d at the periphery of grain e. To show more clearly the effects of the boundary migration, the specimen was lightly polished and then deformed for three additional cycles to a total of 76 cycles. The result is shown in Fig. 3c, from which it is clear that the four grains labelled a to d in Fig. 3a have been consumed by the migration process and the grain labelled e has increased in size especially at the expense of grain f.

Figs. 3d and e show the same area after totals of 126 and 426 cycles, respectively. Again, there is very extensive migration after 426 cycles, and the new boundary positions are revealed more clearly by lightly polishing and deforming for three additional cycles to a total of 429 cycles. The result is shown in Fig. 3f and it confirms again that the grain labelled e in Fig. 3a continues to grow at the expense of grain f.

The same area is shown in Fig. 3g after 729 cycles and in Fig. 3h after lightly polishing and deforming through three more cycles to 732 cycles. In this condition, the grain initially labelled e in Fig. 3a now occupies a major portion of the field of view. It appears also that, at least qualitatively, there is a decrease in the rate of migration with increasing numbers of cycles. This may be appreciated by comparing the large amounts of migration which occur between Figs. 3a and c, representing a change from 3 cycles to only 76 cycles, and the relatively smaller amounts of migration occurring between Figs. 3f and h, corresponding to a change of over 300 cycles from 429 to 732 cycles, respectively.

There are also two additional points of interest with respect to Fig. 3h. First, there is some evidence for the gradual appearance of the orthogonal grain configuration after ~ 700 cycles, and this qualitative observation is supported by the detailed angular measurements described in the following section. Second, when the rate of migration of the grain boundaries slows down at the higher values of N, there is evidence for the division of the grains into smaller subgrains as shown, for example, in the large grain in the upper right of Fig. 3h.

The final photomicrograph of the sequence, shown in Fig. 3i, was taken after a total of 1132 cycles. A comparison with Fig. 3h, taken 400 cycles earlier, shows that there is very little additional migration so that the grain boundaries have almost stabilized into their final configuration. It is clear that many of the boundaries now tend to lie close to the orthogonal configuration and, furthermore, that deformation is confined mainly to the development of a subgrain network within the grains. Subgrains were visible within essentially all of the grains examined by surface microscopy after 1132 cycles and a very clear example is shown in the large grain at the upper left of Fig. 3i. A further qualitative observation, not supported by detailed measurements, was that the subgrain boundaries also tend to exhibit an orthogonal configuration so that many of the subboundaries lie almost parallel to the grain boundaries.

3.2. Angular distributions of the grain boundaries

A second specimen was tested to provide histograms of the angular distributions of the grain boundaries with respect to the stress axis. The results are shown in Fig. 4, where θ is the angle between the surface trace and the longitudinal axis of the specimen, the individual datum points are collected into 10° increments, and the results are expressed as a percentage so that the total area below each line is a constant.

The angular distribution in the untested condition at $N = 0$ is shown in Fig. 4a. As anticipated, the distribution is essentially uniform after the annealing treatment but without the application of any loading cycles: similar results were reported earlier for copper [17, 21] and a copper alloy [17], α-Fe [22] and lead [21] in the annealed condition.

The specimen was tested at 573 K with a strain amplitude of $\pm 0.25\%$ and a frequency of 3.3×10^{-2} Hz. Fig. 4b shows the angular distribution after 100 cycles, and it is apparent already that the very extensive migration occurring in the early stages of testing, as shown in Figs. 3c and d, is leading to a preferential alignment of the grain boundaries into the orthogonal or 45° positions.

The trend is more obvious after 800 cycles, as shown in Fig. 4c where the numbers of boundaries at the very small (<10°) and very large (>80°) angles has decreased markedly. This result is consistent with the qualitative implications of the photomicrograph shown in Fig. 3h.

Finally, Fig. 4d shows the angular histogram after 2500 cycles, and at this point it is clear that a large proportion of the grain boundaries (~25%) lies in the anticipated 40 to 50° increment associated with the orthogonal grain configuration. This result is similar to reports on OFHC copper [17] and α-Fe [22] that ~30% of the boundaries lie in the 40 to 50° range after fatigue testing to failure.

4. Discussion

4.1. General observations

The initial impetus for this investigation was to provide a direct link between the various observations of cyclic grain boundary migration in high temperature fatigue after very small numbers of cycles (up to ~150 cycles) and the numerous

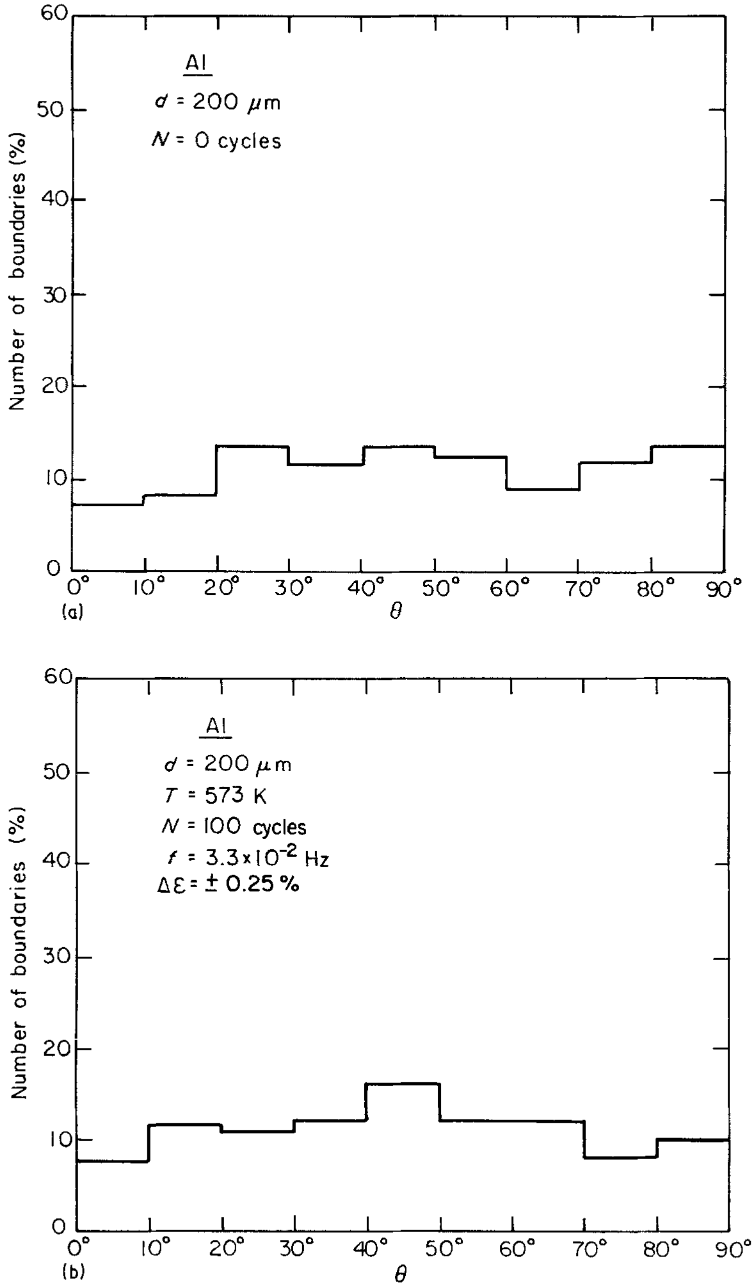
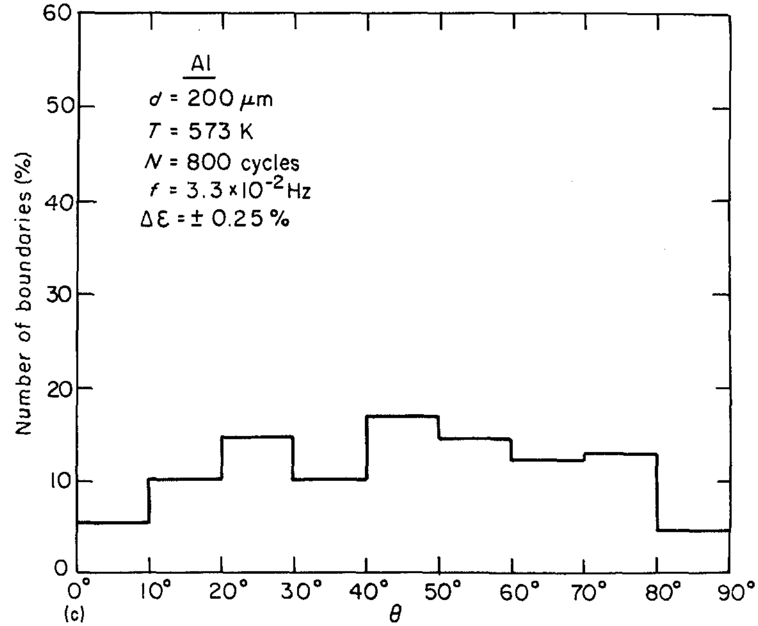


Figure 4 Distributions of the angle, θ, between the surface traces of the grain boundaries and the stress axis for aluminium tested at 573 K with a strain amplitude of ± 0.25% and a frequency of 3.3×10^{-2} Hz: after (a) 0 cycles, (b) 100 cycles, (c) 800 cycles and (d) 2500 cycles.



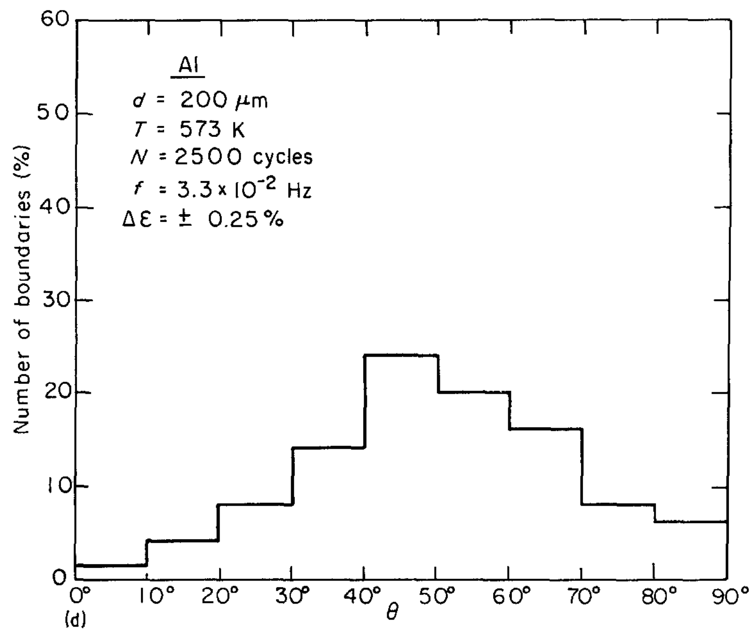


Figure 4 Continued.

reports of an orthogonal grain configuration after large numbers of cycles ($>10^4$ cycles).

There are four important observations arising from this study, and these may be summarized briefly as follows:

1. The cyclic migration observed in the very early stages of fatigue testing leads, ultimately, to the development of an orthogonal grain configuration. There is very extensive migration at the beginning of the test, but the amount of migration tends to decrease at higher numbers of cycles.

2. The process of migration may lead to grain growth. On the one hand, small grains may disappear when boundaries migrate across the widths of the grains; on the other hand, some small grains may increase in size at the expense of their larger neighbours when the boundaries migrate in an outwards direction. The sequence of photomicrographs shown in Fig. 3 gives examples of both types of behaviour.

3. As the rate of migration slows down at the higher numbers of loading cycles, the grains tend to become divided into subgrains. In aluminium of 99.99% purity tested at 573 K with a strain amplitude of ± 0.25% and a frequency of 1.7×10^{-2} Hz, the division into subgrains becomes visible from surface observations after about 700 loading cycles. The subgrain boundaries tend also to exhibit an orthogonal configuration.

4. There is qualitative evidence for the development of the orthogonal grain configuration after ~ 800 loading cycles, and this trend is supported by detailed measurements of the angular distributions. Measurements on aluminium show that a large proportion of the grain boundaries (~ 25%) is in the 40 to 50° angular range after testing through 2500 cycles at 573 K with a strain amplitude of ± 0.25% and a frequency of 3.3×10^{-2} Hz.

The significance of these various observations is considered in the following sections.

4.2. The role of cyclic migration

The cyclic migration observed in the early stages of fatigue testing at high temperatures represents the initial movements of the grain boundaries towards the orthogonal configuration. This is consistent with, and confirms, the assumption incorporated into topological models of the migration process [7, 9].

The observation that the migration occurs more extensively in the early stages of the test is consistent with experimental measurements of the average amounts of migration, $\bar{m}$, as a function of the number of loading cycles, N. Snowden *et al.* [42] showed that, for aluminium of 99.99% purity, copper, zirconium and Zircaloy-2, a logarithmic plot of $\bar{m}$ against N gave a linear relationship up to $N \simeq 10^4$ cycles, equivalent to ~10% of the total fatigue life, and thereafter there was no increase in the amount of migration. For $N < 10^4$ cycles, it was reported that

$$\bar{m} \propto N^{0.65} \tag{3}$$

for all four materials. More recent work [43], also on aluminium of 99.99% purity, gave

$$\bar{m} \propto N^{0.5}. \tag{4}$$

These results confirm, therefore, the decrease in the rate of migration at the higher numbers of loading cycles, and the observations of Snowden *et al.* [42] suggest that the orthogonal grain configuration is in a final stabilized form in aluminium of 99.99% purity after ~ 10^4 cycles. The latter conclusion is consistent with the angular distributions shown in Fig. 4.

4.3. The possibility of a change in grain size

There is no doubt that the grain size often changes during fatigue testing at high temperatures but the precise trend is not clearly defined. In early work, grain growth was reported during the fatigue testing of Mg–0.8% Al [25, 44] whereas there was a decrease in the average grain size of copper due to recrystallization [18]. Very recently, Snowden *et al.* [20] showed that the grain size of copper may either increase or decrease during fatigue testing depending only on the initial grain size: specifically, it was shown that the grain size increases when testing with an initial grain size of 35 μm, it decreases with an initial grain size of 400 μm, and in both materials the ultimate equilibrium grain size is of the order of ~ 130 μm.

The present results, as given in the series of photomicrographs in Fig. 3, show that grain growth occurs in aluminium under the present testing conditions, and the average grain size increases by the elimination of some (*but not all*) of the smaller grains. This is consistent with an early observation of the elimination of the smaller grains during fatigue testing of aluminium of similar purity [13]. It appears also from Fig. 3i that the formation of a subgrain structure may lead, ultimately, to the break up of the larger grains, in a manner analogous to the observation by Snowden *et al.* [20] that new grains are formed in the periphery of grains of copper with a large initial grain size.

4.4. The role of subgrain formation

In the present work, the formation of subgrains was observed after testing through large numbers of cycles (≳ 700 cycles, as shown in Figs. 3h and i). Westwood and Taplin [22] reported subgrain formation in α-Fe and they noted also, as in the present work, that the subgrain boundaries tend to become aligned with the grain boundaries making up the orthogonal grain configuration.

It was suggested by Westwood and Taplin [22] that the preferential alignment of the sub-boundaries is associated with the nature of the slip occurring within the grains, since the dislocations will build up on those planes experiencing the maximum shear stresses and the climb of these dislocations to form low angle sub-boundaries perpendicular to the slip planes will develop the orthogonal configuration. This suggestion is supported in part by the early observation of Snowden [29] on high purity lead that the majority of slip traces (~ 65%) lie within the angular range of $\theta = 30$ to $60°$.

An additional implication from the present investigation is that subgrain formation takes place primarily when the grain boundaries have essentially stabilized into the orthogonal grain configuration: prior to stabilization, the migrating boundaries sweep out large areas of many of the grains so that subgrain formation is not possible in the early stages of testing. Inspection shows that this suggestion is consistent also with the data of Westwood and Taplin [22] on α-Fe, where subgrain formation was reported after 125 cycles, the angular distribution after 125 cycles was similar to the distribution at fracture with ~ 30% of the grain boundaries in the 40 to 50° range, and the specimen ultimately failed after only ~ 200 loading cycles.

4.5. The significance of the orthogonal grain configuration

The sequence of photomicrographs shown in Fig. 3 and the angular distributions shown in Fig. 4 both confirm that the grain boundaries gradually move into, and stabilize at, the orthogonal grain configuration. This is consistent with the experimental observation by Snowden *et al.* [42] that migration ceases in aluminium after ~ 10^4 cycles. It is qualitatively consistent also with the model of Wigmore and Smith [2] in which sliding on boundaries close to the 45° orientations produces strains at the triple points and consequent migration of other boundaries into the 45° positions in order to reduce the strain energy.

In practice, experimental data suggest that the rate of attaining the orthogonal configuration depends upon both the strain amplitude and the purity of the material. Snowden [21] reported angular measurements on lead in which there was a marked increase in the proportion of boundaries lying in the 40 to 50° range when the strain amplitude was increased by about an order of magnitude*. Saegusa and Weertman [19] performed reverse bending tests on copper 99.999% and 99.9% purity, and they showed by angular measurements that, when both materials were tested at the same temperature (678 K), the same frequency (16.7 Hz) and similar strain amplitudes (± 0.035% and ± 0.03% for the high and low purities, respectively), the orthogonal configuration was attained in the high purity copper after 3×10^4 cycles whereas there was only a slight movement towards the orthogonal structure in the copper of lower purity after 7.2×10^5 cycles.

Finally, it is important to note that the development of the orthogonal grain configuration has important implications when considering the role of cavitation in fatigue. Experiments on copper [16, 19] and Mg–0.8% Al [16] have shown that the level of cavitation, representing the proportion of boundaries with visible cavities, is highest (> 60%) on those boundaries lying in the 40 to 50° angular range. This observation confirms the close inter-relationship between grain boundary sliding, grain boundary migration and intergranular cavitation in high temperature fatigue.

5. Summary and conclusions

1. Experiments on polycrystalline aluminium subjected to reverse bending fatigue at a temperature of 573 K show that the cyclic grain boundary migration observed in the very early stages of testing leads ultimately to the development of an orthogonal (diamond) grain configuration. The rate of migration is rapid in the early stages of testing but decreases after large numbers of fatigue cycles.

2. A series of angular measurements demonstrates that the grain boundaries gradually migrate so that a large proportion (~ 25%) lies in the angular range of 40 to 50° to the stress axis.

3. The presence of boundary migration may lead to grain growth. Some small grains may be eliminated when boundaries migrate across their widths, whereas other small grains may increase in size when migration occurs in an outwards direction.

4. As the rate of migration slows down after large numbers of testing cycles, the grains become divided into subgrains. The subgrain boundaries tend also to exhibit an orthogonal configuration.

*The measurements by Snowden [21] were obtained after testing through ~ 10^5 to 10^7 cycles at strain amplitudes of ± 0.016% and ± 0.140%, respectively. Although there is a clear tendency for the orthogonal grain configuration to be reached more rapidly when the strain amplitude is increased, this trend probably breaks down at large strain amplitudes since it was shown for aluminium that there is essentially a negligible change in the amount of migration occurring in each cycle of loading at strain amplitudes above about ± 0.4% [43].

Acknowledgement
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