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On the formation of the diamond grain configuration during high temperature creep and fatigue

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A study has been made of the influence of test variables on the formation of the diamond grain configuration during high temperature creep and fatigue deformation of a wide variety of metals. The proposed mechanisms for the formation of this interesting grain morphology are reviewed. It is concluded that the diamond grain configuration arises from a balance between grain-boundary sliding, grain-boundary mobility, intragranular deformation and defect imbalance across the grain boundaries and that it tends to be stabilized by intergranular cavitation. While the phenomenon occurs during high temperature fatigue in a variety of metals irrespective of their crystal structure, during creep it has been observed only in to h c p metals. It is surmised that the occurrence of the diamond array of grain boundaries during creep deformation in h c p metals is aided by the limited number of slip systems which leads to high defect imbalances in adjacent grains and consequently high driving forces for grain-boundary migration. On the basis of quantitative metallography involving measurements of the number of edges per grain section, the number of grains meeting at vertices, angular distribution histograms and grain-boundary lengths in different angular orientations with respect to the stress axis in "annealed" and "diamond" microstructures, it is concluded that the shape of the "diamond" grain is essentially the same as that of the "annealed" grain but in a distorted form.

1. Introduction

The formation of a diamond grain configuration (DGC), in which a majority of grain boundaries become aligned at about 45° to the stress axis on longitudinal sections without a significant change in grain size, is a striking microstructural development during deformation of metals at temperatures about about $0.5T_m$ (T_m is the absolute melting temperature). Many metals, namely Pb [1–4], Cu [5–8], Ni [9], Al [10], Mg [11], Cd [12], Zr [13], Fe [14, 15] and alloys such as austenitic stainless steel [16] have been reported to exhibit such a grain morphology during high temperature fatigue. No report of a similar observation has been made during monotonic deformation at elevated temperatures except for the recent work on zinc by Singh *et al.* [17] and on cadmium by Sastry *et al.* [12]. Thus, the prevailing view that the DGC is characteristic only of high temperature cyclic deformation needs to be

modified to include high temperature monotonic deformation in certain instances. Various important parameters such as grain-boundary sliding [18-20], creep cavitation [17, 19], creep strength [19, 21] and fatigue life [15, 22–24] are affected by the DGC.

In view of the general formation of the DGC during high temperature fatigue but only rarely during high temperature monotonic deformation, this study was undertaken to examine the effect of strain, strain-rate, temperature, grain size and crystal structure of the material on the grain-boundary configurational changes during creep and fatigue deformation. Quantitative metallography has also been used to analyse the shape of the solid grain that appears as a diamond grain on longitudinal sections.

2. Experimental

Tensile tests were carried out on a Zn–0.14 at.% Cu alloy using an Instron machine modified for use at elevated temperatures. This alloy shows an increasing tendency to form the DGC with increasing temperature when tested in tension at a nominal strain-rate of $0.01\,h^{-1}$ and reaches a maximum at 100° C (0.54 T_m) [17]. Thus, the effect of other variables such as stain-rate, strain and grain size was studied by testing this alloy at 100° C and strain-rates of 0.01, 0.1 and $3.60\,h^{-1}$ to total strains of up to 32%. Specimens with initial equi-axed grain sizes of 120 and 750 μm were also compared.

To examine the influence of crystal structure on the formation of the DGC, the h c p Zn–Cu alloy and f c c Al were deformed in creep as well as low cycle fatigue at elevated temperatures. Low cycle fatigue tests have also been undertaken on OFHC copper (f c c) at temperatures between 300 and 600° C (0.42 to 0.64 T_m) and on pure iron (b c c) at temperatures between 500 and 700° C (0.43 to 0.54 T_m). All specimens were quenched with water or compressed air after testing and control experiments, similar to those described elsewhere [23], were undertaken to ensure that these procedures retained the structure developed at the test temperature.

Longitudinal sections of the creep and fatigue-tested specimens were prepared using conventional metallographic techniques. The angular distribution of grain boundaries was determined by measuring the angle between the stress axis and the tangent to the trace of the boundary on the sectioned surface. The boundaries were sampled by measuring all the boundaries in a given area until a total of 200 was reached. The effect of intergranular caviation on the DGC in creep-tested Zn–Cu specimens was investigated by analysing the uncavitated boundaries and also the total boundaries (including catitated boundaries).

The shape of the solid grain showing the DGC was analysed by quantitative metallography. Longitudinal and transverse sections and sections at 45° to the stress axis were prepared from both annealed and tested Zn–Cu specimens which showed maximum tendency for the DGC. Micrographs were taken from the longitudinal section and the number of edges for each grain was counted. A total of 200 grains was analysed using the same sampling procedure as described above. The total length of grain boundaries as a function of their orientation relative to the stress axis was measured with micrometers on the microscope stage. This technique was found to be sufficiently accurate for measuring the lengths of very short grain boundaries. The orientation of each grain boundary to the stress direction was also measured from enlarged micrographs. The results were presented as histograms.

3. Results and discussion

3.1. Proposed mechanisms for fomation of diamond grain configuration

Snowden [1] was the first worker to report the development of a DGC during fatigue. He attempted to correlate the preferential orientation of grain boundaries at 45° to the stress axis with intragranular deformation in Pb, fatigued at room temperature (0.5T_m), by measuring the angular distribution of slip traces and found that a majority of slip traces were oriented near ±45° to the stress axis. In a later investigation using bicrystals of Pb, Snowden [3] made an important observation that serrations developed in the grain boundary parallel to active slip planes before fatigue-hardening was complete. The formation of grain-boundary serrations has been attributed to reciprocal migration arising from an imbalance of dislocation pressures on either side of the boundary.

Wigmore and Smith [7] have suggested a model for the development of the DGC during high temperature fatigue of copper which emphasizes the role of grain-boundary sliding. They suggest that ahead of the boundaries already at 45° positions,

high local strains are generated by the higher shear stresses acting on these boundaries as compared to adjoining boundaries which are not initially at 45° orientations. Thus, non-uniform deformation in neighbouring grains generates a defect imbalance which is minimized by migration of grain boundaries towards 45° orientations. The concept of strain-induced grain-boundary migration during high temperature deformation, based on an imbalance of dislocations across the boundaries arising from inhomogeneous deformation in neighbouring grains, has been proposed earlier by a number of investigators [5, 25–27]. The *in situ* observations made by Walter and Cline [20] using a high speed photographic technique during the creep deformation of Al illustrate the interrelationship between grain-boundary sliding, the resulting shear zones and migration of boundaries to 45° positions. The importance of grain-boundary sliding in forming the DGC during hot deformation is also emphasized in the recent work on cadmium [12] and in the work by Puccine [28] on the torsional fatigue of copper.

Alpha iron fatigued at 700° C develops sub-boundaries at 45° positions with grain boundaries aligned parallel to the sub-boundaries [15, 19]. It is suggested that some intragranular slip arises from the requirement to accommodate sliding at triple junctions; dislocations build up on planes experiencing maximum resolved shear stress and then climb to form low angle boundaries, associated with which is a surface tension and defect imbalance driving grain boundaries to become aligned parallel to the sub-boundaries.

In recent work [17], where the first observation of a DGC during creep is presented, the formation of subgrains or cells in a Zn–0.14% Cu alloy has been noted. Cell formation is not uniform in all the grains because of differences in grain orientation with respect to the stress axis. A source of grain-boundary migration perhaps relevant here is the unbalanced pull exerted by the subgrains on one side of a grain boundary when these are more numerous than those on the other side. Texture in the material is not associated with the DGC [17].

In summary, the processes which appear to play an important role in the formation of the DGC during formation at high temperatures are grain-boundary sliding, intragranular deformation, defect imbalances across boundaries, substructure formation and grain-boundary migration. An examination of the effect of test variables during high temperature creep and fatigue is expected to provide some indication of the role of these processes in the formation of the DGC. The influence of crystal structure on the formation of the DGC during creep is also considered in view of its appearance only in h c p metals to date.

3.2. Effects of test variables during creep of Zn–0.14% Cu alloy on the diamond grain configuration

3.2.1. Effect of temperature

Fig. 1 shows the variation of relative frequency of uncavitated and total boundaries in the 30 to 60° range with test temperature for an initial grain size of 230 μm. An increasing number of uncavitated boundaries have aligned at ~45° to the stress axis as the temperature is raised to 100° C ($0.54T_m$). Beyond this temperature the uncavitated boundaries migrate out of the DGC. On the other hand, caviation that develops with increasing temperature has a pinning influence on the grain boundaries and so stabilized the DGC.

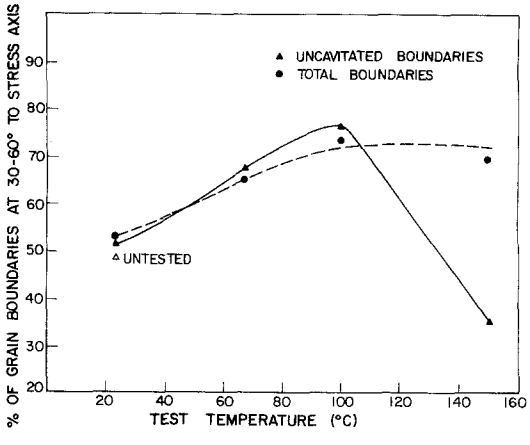

Figure 1 Variation in number of uncavitated and total grain boundaries lying in the angular range 30 to 60° to the stress axis with test temperature in Zn–0.14% Cu alloy, fractured in slow tension (strain-rate 0.01 h^{-1}).

3.2.2. Effect of strain-rate

The histograms in Fig. 2a, b and c show the angular distribution of grain boundaries with respect to the stress axis on longitudinal sections of Zn–Cu specimens tested at 100° C and strain-rates of 0.01, 0.1 and 3.60 h^{-1} respectively. The DGC is pronounced at a strain-rate of 0.01 h^{-1} but at the higher strain-rates, the DGC is almost insignificant.

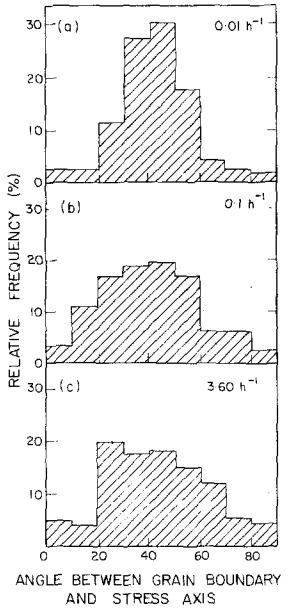

Figure 2 Histograms showing angular distribution of grain boundaries with respect to the stress axis for Zn–0.14% Cu specimens tested at 100° C at strain-rates of: (a) 0.01 h^{-1}, mean = 42°, standard deviation (S.D.) = 14°; (b) 0.1 h^{-1}, mean = 41°, S.D. = 19°; (c) 3.60 h^{-1}, mean = 44°, S.D. = 19°.

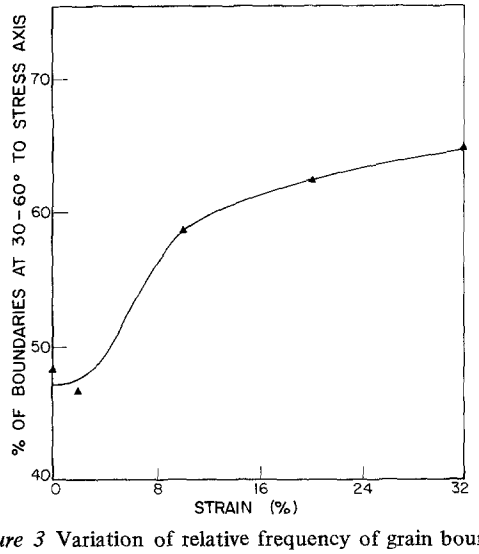

Figure 3 Variation of relative frequency of grain boundaries lying in the angular range 30 to 60° to the stress axis in Zn–0.14% Cu alloy as a function of creep strain (test temperature 100° C; strain-rate 0.01 h^{-1}).

3.2.3. Effect of strain

Fig. 3 shows the variation of relative frequency of grain boundaries in the angular range 30 to 60° with respect to the stress axis as a function of creep strain. There is a rapid increase in the percentage of grain boundaries in the angular range 30 to 60° with increasing strain up to about 10% and above this strain the rate of incease is relatively much slower. An insignificant change in the orientation of grain boundaries below about 4% strain is also noted.

3.2.4. Effect of grain size

Fig. 4a and b show the angular distribution of grain boundaries for specimens with initial grain sizes of 120 and 750 μm respectively and tested under identical conditions. There is a marked tendency for preferential alignment of grain boundaries at about 45° to the stress axis only in the fine grained specimen.

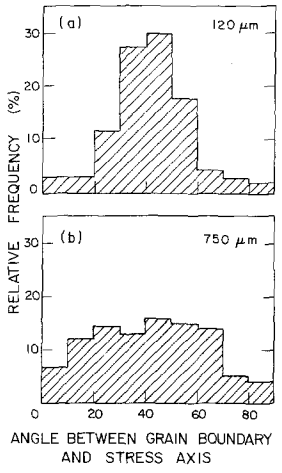

Figure 4 Histograms showing angular distribution of grain boundaries in Zn–0.14% Cu alloy for grain sizes of: (a) 120 μm, mean = 42°, S.D. = 15°; (b) 750 μm, mean = 42°, S.D. = 21°. (Test temperature 100° C; strain-rate 0.01 h^{-1}; strain 10%).

3.2.5. Effect of solute addition

Addition of a solute which segretates to the grain boundaries, such as Al in Zn, can cause a marked reduction in grain-boundary mobility and so inhibit the formation of the DGC during high temperature creep [17].

3.2.6. Discussion

The results show that during creep of a Zn–0.14% Cu alloy, intermediate temperatures, low strain-rates, strains beyond a critical level and finer grain sizes favour the formation of the DGC. At temperatures below about $0.4T_m$ grain-boundary sliding and grain-boundary migration do not occur. When grain boundaries are pinned by solutes (e.g. Al in Zn), the DGC does not form.

The importance of grain-boundary sliding in the formation of the DGC is reflected in its formation at lower strain-rates. That a certain creep stain must be exceeded for the reorientation of grain boundaries suggests the need to build up a threshold defect imbalance. The stabilization of the DGC at higher strains could be due either to the onset of cavitation, substructure formation or a lowering of the defect imbalance across the moving boundaries as they migrate close to 45° orientations. The absence of the DGC in the coarse grained specimen is probably due to intragranular deformation (accompanying grain-boundary sliding) being highly localized and to a large migration distance required to produce the DGC. It is also relevant here that Gifkins [26] has shown that the activation energy for grain-boundary migration during creep of Pb is higher for coarse grained specimens than for fine grained ones.

It therefore appears that the DGC arises during creep from an appropriate balance between grain-boundary sliding, intragranular deformation and defect imbalance across the moving boundaries and is most pronounced when the migration distance is low. These points are discussed further in a later section.

3.3. Effects of test variables during high temperature fatigue on the Diamond Grain Configuration

3.3.1. Effect of temperature

The influence of test temperature on the development of the DGC during fatigue of alpha iron [15] and OFHC copper is summarized in Table I. All data were obtained from specimens tested to failure. These results show that little migration occurs at the lower temperatures but that at 650 to 700° C (0.51 to $0.54T_m$) for Fe and 450 to 600° C (0.53 to $0.64T_m$) for Cu there is a marked tendency for boundaries to align preferentially at about 45° to the stress axis. Thus, the test temperature has a marked influence on the extent of grain-boundary reorientation during low cycle fatigue and the DGC in these materials in promoted by temperatures $\geqslant 0.5T_m$.

3.3.2. Variation with number of fatigue cycles

Fig. 5 shows the relationship between relative frequency of grain boundaries in the angular range 30 to 60° to the stress axis and fraction of fatigue life for high purity Al tested at 375° C ($0.70T_m$) and 85 mHz. A rapid rise is observed in the relative frequency of boundaries in the angular range 30 to 60° up to about 7% of the expected fatigue life, beyond which there appears to be a tendency towards saturation. Similar observations on the rapid formation and stabilization of the DGC during early fatigue life have been made earlier on Fe [15] and recently on OFHC Cu [23]. Table II summarized the results for these materials. An important observation from these studies is that the onset of intergranular cavitation coincides with a stabilization of the DGC.

TABLE II Variation of percentage of grain boundaries at 30 to 60° to the stress axis with number of fatigue cycles (expressed in terms of number of cycles to failure, N_f) for Fe [15] and Cu [23]

Number of cycles	Fe Nominal plastic strain ± 2% 85 mHz, 700° C	Cu Nominal plastic strain ± 0.7%, 58 mHz, 490° C
Annealed	33	32
$0.05N_f$	55	50
$0.10N_f$	47	53
$0.20N_f$	57	55
$0.50N_f$	67	62
Fracture	64	61

TABLE I The effect of fatigue temperature on the percentage of grain boundaries oriented at 30 to 60° to the stress axis in Fe (after Westwood and Taplin [15]) and Cu

Fe Nominal plastic strain ± 2%; 85 mHz		Cu Nominal plastic strain ± 0.7%; 58 mHz	
Temperature (° C)	% of boundaries at 30 to 60° to stress axis	Temperature (° C)	% of boundaries at 30 to 60° to stress axis
Annealed	33	Annealed	32
500	31	300	35
550	41	400	35
600	37	450	56
650	48	490	61
700	64	600	66

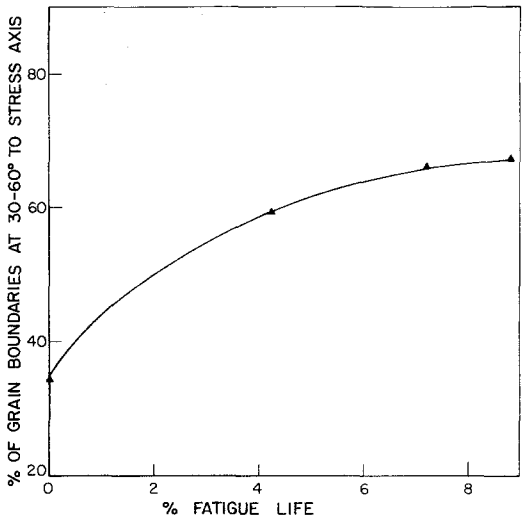

Figure 5 Variation of relative frequency of grain boundaries in high purity Al lying in the angular range 30 to 60° to the stress axis as a function of fatigue life (temperature 375° C; nominal strain amplitude ± 2%, frequency 85 mHz).

A small load drop of about 5% was observed in the load response curve during the early stages of low cycle fatigue tests on Al at 375° C. Load drops have also been reported for other metals tested under similar fatigue conditions [15, 23]. It is precisely during the same initial stage that marked grain-boundary migration to the DGC takes place. The boundaries at 45° to the stress axis experience maximum shear stress which could lead to increased grain-boundary sliding that may be responsible for the observed softening. An inverse dependence of the magnitude of the drop load on stain amplitude has been found in alpha Fe [29] and it is pointed out as evidence that the softening is due to grain-boundary sliding since the sliding contribution is likely to be less at higher applied strains due to the need for accommodation at triple points.

3.3.3. Effect of cycle frequency

The formation of the DGC has been reported in low cycle fatigue [7, 8, 10, 15, 16] as well as high cycle fatigue [5, 11, 12] at high temperatures. However, during low cycle fatigue at constant strain amplitude, the rate at which the DGC if formed appears to depent on cycle frequency. This effect has been examined [29] in terms of an arbitrary parameter N_{75}, defined as the number of cycles required for 75% load drop from initial to steady load. The N_{75} parameter has been shown to decrease with decreasing frequency.

3.3.4. Effect of grain size

Limited studies on Al [25] and alpha Fe [29] have revealed that the DGC is significant in fine grained specimens but absent in coarse grained specimens after low cycle fatigue at high temperatures.

3.3.5. Discussion

During high temperature low cycle fatigue, the DGC forms within about 5% of the expected fatigue life and is favoured by increasing temperature, lower frequency and finer grain size. With increasing temperature the contribution of grain-boundary sliding during fatigue increases, as does the grain-boundary mobility and the rate of climb of dislocations to form sub-boundaries [29]. However, data regarding the formation of the DGC and the extent of grain-boundary sliding at temperatures beyond $0.7T_m$ are lacking and further work is necessary to establish the effect of very high grain-boundary mobility. The rapid formation of DGC in pure metals during the first few cycles is attributed to the high rate of grain-boundary mobility and grain-boundary sliding in the initial stages of fatigue.

Less pure metals [7, 9] and complex alloys [23, 30], in which grain-boundary migration is drastically reduced by the pinning influence of solutes and complex phases, generally, do not show the DGC. The significance of grain-boundary migration in the formation of DGC has recently been demonstrated by Cocks and Taplin [23]. They have shown that a commercial copper alloy, CDA 195, fatigued at 300° C exhibits the required cell morphology and orientation for the formation of DGC according to earlier suggestions [15, 19] but the alloy does not show any tendency for preferential alignment of boundaries at 45° to the stress axis because of low grain-boundary mobility at this temperature. At higher temperatures, where there was a tendency to develop DGC, the cells were very coarse and most of the grains did not contain cells. These observations suggest that the alignment of grain boundaries to 45° positions requires adequate grain-boundary mobility and that the strain-induced migration depends mainly on a defect imbalance across the grain boundary and not on the cell morphology. The early stabilization of the DGC may be due to the reduction of

defect imbalance with the formation of DGC, the onset of cavitation and also because there is no net grain elongation in fatigue. Lower fatigue frequencies in constant strain amplitude tests result in lower strain rates so the tendency for the formation of the DGC at low frequencies can be understood in terms of greater time available for the operation of thermally activated processes. The absence of DGC in coarse grained material even during fatigue is attributed to the large migration distance involved.

3.4. Crystal structure and the diamond grain configuration

Specimens of Zn–Cu tested in low-cycle fatigue at 100° C were found to exhibit a very pronounced DGC. As mentioned in Section 1, a variety of metals irrespective of their crystal structure form the DGC during high temperature fatigue. To date, only two h c p metals, Zn [17] and Cd [12], have been reported to develop a DGC during creep deformation. In order to study the effect of crystal structure on the formation of DGC during creep deformation, polycrystalline Al (f c c) specimens were tested in slow tension at 250° C. The boundaries tended to align at about 45° to the stress axis on the surface of the specimens (Fig. 6a) but the DGC was not detected below the surface (Fig. 6b). It has been reported elsewhere that Al tends to form the DGC only during low cycle high temperature fatigue [10] but not during creep [31–33]. Both Cu (f c c) and alpha iron (b c c) also show a strong tendency to form the DGC during high temperature fatigue but do not show any evidence for DGC during creep deformation [23, 29].

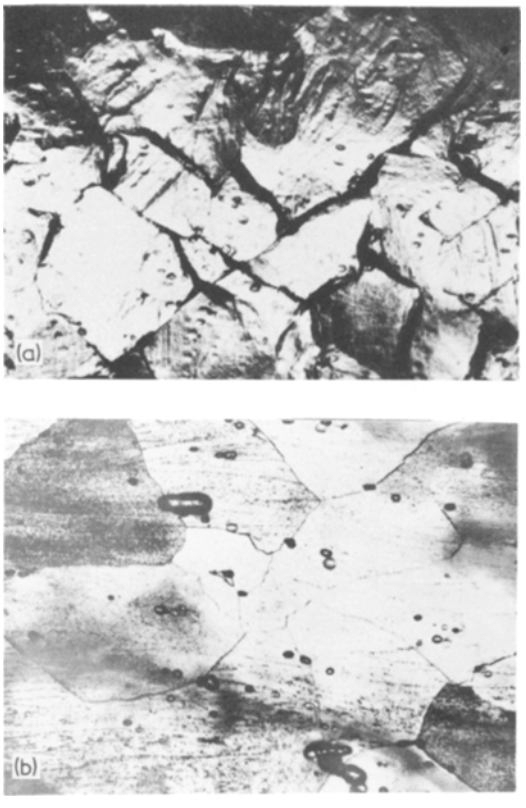

Figure 6 Micrographs from high purity Al teste in creep: (a) specimen surface; (b) longitudinal section. (Test temperature 250° C; nominal strain-rate 0.01 h^{-1}.) Stress direction horizontal. × 100

These results, indicating that Cd and Zn, but not Al, Cu and Fe develop a DGC during creep whereas a variety of metals, irrespective of crystal structure, exhibit the DGC during high temperature fatigue may be discussed by comparing the mechanisms of high temperature creep and fatigue deformation.

A point of major importance is that the defect imbalance during fatigue may arise due to the higher dislocation density, observed for example by Challenger and Moteff [34] in transmission electron microscopy of AISI 304 and 316 stainless steels. Interestingly, Feltner [35] has noted that repeated stressing only in tension caused no difference in dislocation density of Al specimens and has concluded that reversed stressing is necessary to generate greater defect densities.

Apart from higher dislocation density, a much higher concentration of lattice vacancies is also generated during fatigue than during creep [6, 36–38]. The enhanced supply of lattice vacancies, which arrive at the grain boundaries probably by pipe diffusion [38], could also contribute to greater grain-boundary mobility under fatigue conditions. This rapid grain-boundary mobility is the major reason for easy and very rapid formation of the DGC in the early stages of fatigue. The early stabilization of the DGC during fatigue is probably due to the rapid decrease in defect imbalance across the boundaries as they migrate towards 45° orientations. The stability is further aided by intergranular cavitation along 45° boundaries. The formation and growth of cavities is much faster during high temperature fatigue than during creep deformation under similar testing conditions [6].

It is also possible that the "diamond" grains do not develop during creep of f c c and b c c metals

because grain elongation in the stress direction prevents the boundaries stabilizing at 45° positions (by comparison there is no net elongation in fatigue and creep tested c p h metals exhibit low grain elongations). A concept that needs to be considered further in this context is that of grain rotation which can lead to considerable lessening of the grain elongation in the direction of the applied stress [39].

In view of the importance of defect imbalance in grain-boundary migration, it is likely that the number of available slip systems has a major effect on the formation of the DGC during creep. The smaller number of slip systems in the h c p metals (Zn, Cd), as compared to the f c c (Cu, Al) and b c c (alpha Fe) metals, may give rise to a situation where the ease of deformation in adjacent grains is markedly different because of the different grain orientations. Thus, a greater defect imbalance may be developed across the grain boundaries during creep of h c p metals. The reason for the occurrence of the DGC on the surface of creep tested Al specimens and its absence in longitudinal sections requires further investigation, but the observation does point to an important role of vacancies in the formation of the DGC. The importance of grain-boundary sliding in the occurrence of the DGC at finer grain sizes and lower strain-rates has already been mentioned. It is relevant here to refer to a statement of Bell and Langdon [40] – "it does appear that sliding is of greater importance in h c p metals, presumably because of the limited number of slip systems which are then available for grain deformation". For example, under similar conditions of temperature, stress and grain size, the value of γ (the ratio between strain due to grain-boundary sliding and total strain) is 40 to 80% in h c p Magnox (Mg–0.78 wt% Al alloy) while it is less than 10% in Al. The absence of marked grain elongations in h c p metals showing the DGC during creep also implies that γ is high and extensive grain-boundary sliding may give rise to the high defect imbalance required for migration.

3.5. Shape of grains showing diamond grain configuration

Although the development of a "diamond" or "square" array of grain boundaries on longitudinal sections of specimens deformed under high temperature fatigue conditions has been widely reported, there has been no attempt to examine either the details of this configuration revealed on sectioned surfaces or the three-dimensional shape of the grains. Most workers imply that the diamond array shows a predominance of four-sided grains and four-fold junctions.

Fig. 7a and b show typical micrographs from longitudinal sections of annealed and creep tested Zn–0.14% Cu specimens respectively. Each grain is labelled according to its number of edges. Contrary to the earlier concept that the DGC comprises a predominance of four-fold junctions, it is evident from Fig. 7b that the grain junctions are entirely triple junctions. Junctions which appear to be four-fold were resolved at higher magnification as two triple junctions very close to one other. Fig. 8 shows the relationship between relative frequency and number of edges per grain for the annealed and creep tested specimens. The two curves are quite similar and the curve for the creep tested specimen does not show a peak in relative frequency of four-sided grains. These results point to significant similarities between "annealed" and

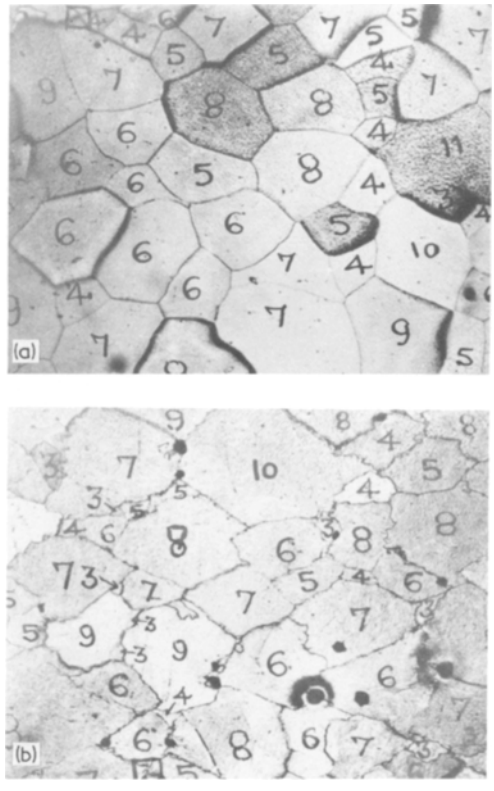

Figure 7 Micrographs of longitudinal sections of Zn–0.14% Cu specimens: (a) annealed; (b) creep tested. (Test temperature 100° C; nominal strain-rate 0.01 h^{-1}; strain 10%). Stress direction horizonal. × 90

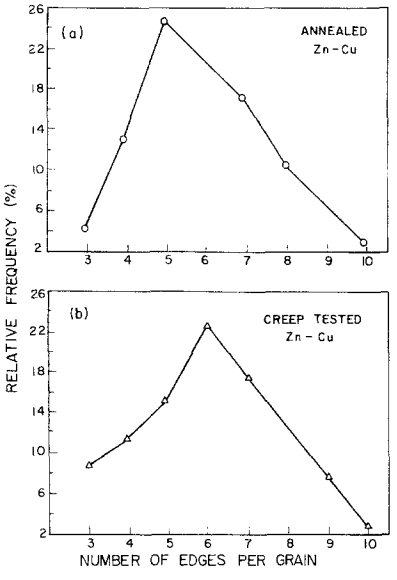

Figure 8 Variation of relative frequency with number of edges per grain on longitudinal section of An–0.14% Cu specimens: (a) annealed; (b) creep tested. (Test temperature 100° C; nominal strain-rate 0.01 h^{-1} ; strain 10%.)

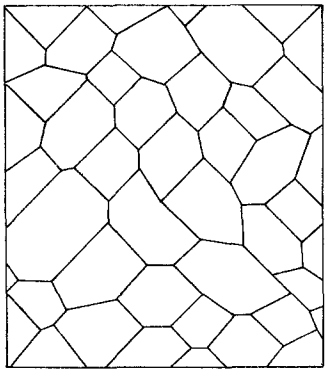

Figure 9 Schematic representation of the diamond grain configuration on a longitudinal section.

"diamond grains". The "diamond" grains are not four-sided nor it there a preponderance of four-fold junctions. A schematic representation of the diamond array is shown in Fig. 9. The average number of edges per grain for the "annealed" body, which has been shown here to be the same for the "diamond" body, has been determined as $5\frac{1}{7}$ [41, 52].

In an attempt to resolved the difference between "annealed" and "diamond" bodies, further investigations were made of the longitudinal sections of the annealed and creep tested Zn–Cu specimens showing the DGC. The angular orientation and length of each grain boundary was

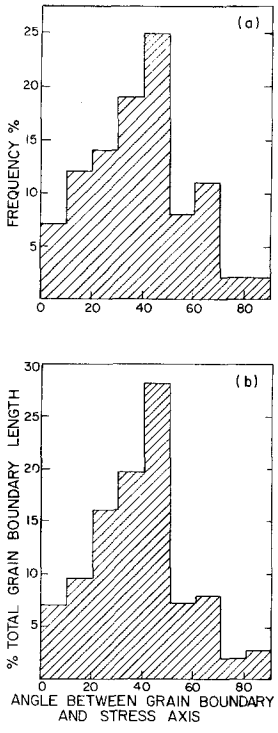

Figure 10(a) Angular distribution of grain boundaries with respect to the stress axis in longitudinal section of creep tested Zn–0.14% Cu specimen, showing prominent DGC. Mean = 38°; S.D. = 19°. (b) Variation of percentage of total length of grain boundaries with angle to the stress axis in the longitudinal section of the same specimen. Mean = 38°; S.D. = 18°.

measured. The annealed specimen showed no tendency for preferential alignment of grain boundaries in any particular orientation and the percentage of the total length of grain boundaries in different angular orientations to the stress axis was also found to be constant. Fig. 10a records the angular distribution of grain boundaries in the creep tested specimen. As expected, the histogram reflects the preferential alignment of grain boundaries at 45° to the stress axis. In addition, a large percentage of the total grain-boundary length lies near 45° to the stress axis (Fig. 10b).

These measurements bring out the dissimilarities between the "annealed" and "diamond" grain. However, in view of the important observation that the number of edges per grain the type of grain junction in the "diamond" array are the same as those in the "annealed" array, it is concluded that the diamond body is merely a distorted annealed body. The distortion is best expressed in terms of the number and length of grain

boundaries lying near 45° to the stress axis on longitudinal sections. Sections taken both transverse and at 45° to the stress axis in the specimen showing prominent DGC in longitudinal sections (Fig. 7b) revealed random orientations of grain boundaries (Fig. 11). Clearly, longitudinal sections are necessary to detect the DGC.

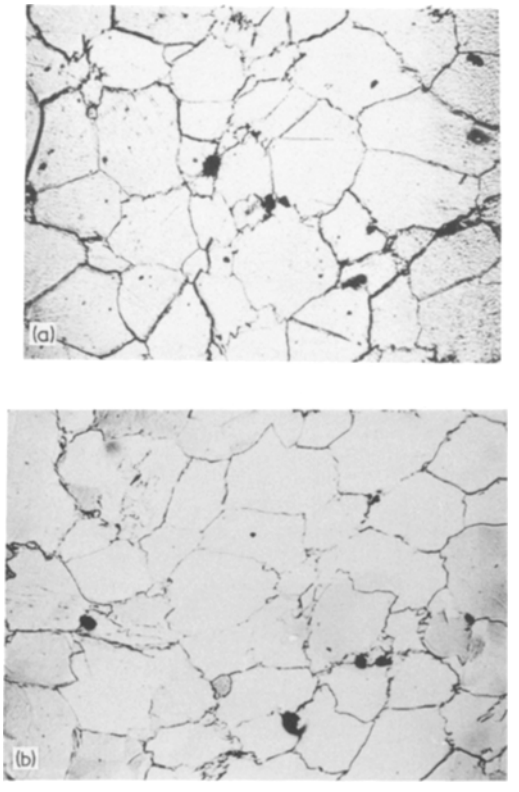

Figure 11 Micrograph of creep tested Zn–0.14% Cu specimen, which showed a prominent DGC on a longitudinal section; (a) transverse section; (b) section at 45° to stress axis. (Test temperature 100° C; strain-rate 0.01 h^{-1}; strain 10%.) × 90

4. Conclusions

(1) The DGC is a characteristic feature of high temperature fatigue of a variety of metals, irrespective of their crystal structure. However, during creep the phenomenon appears to be restricted to h c p metals. The frequent development of the DGC during high temperature fatigue but not during creep is attributed to a higher grain-boundary mobility, arising from both a greater defect imbalance across the boundaries and a higher concentration of lattice vacancies.

(2) An appropriate balance between grain-boundary mobility, grain-boundary sliding, intragranular deformation and defect imbalance across the moving boundaries is necessary for the formation of the DGC. During creep of a Zn–0.14% Cu alloy, intermediate temperatures, low strain-rates, strains above a critical value and a fine grain size favour the formation of the DGC. During high temperature low cycle fatigue, formation of the DGC is favoured by increasing temperature, low frequency and a fine grain size.

(3) Intergranular cavitation tends to stabilize the DGC.

(4) The grains on longitudinal sections showing the DGC are neither four sided nor do they have four-fold junctions. The shape of the "diamond" grain is essentially the same as that of the "annealed" grain but in a distorted form. The distortion is best expressed in terms of the number and total length of grain boundaries oriented near 45° to the stress axis on longitudinal sections.

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