although its complexity indicates that visual observation alone is insufficient for definitive characterisation. Differences in the stress system imposed during essentially uniaxial loading as compared to that existing during unloading may account for the observed differences between loading and unloading transformations. The difference between loading and unloading stress systems would be less significant for nucleation and growth transformations; thus for carbon tetrachloride both loading and unloading characteristics were the same.

Acknowledgements

The authors gratefully acknowledge the encouragement of C. D. Dickinson, and the helpful suggestions of G. Piermarini, A. Van Valkenburg, and H. Margolin.

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23 February 1968, and in revised form 1 June

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On the Formation of Grain-Boundary Cracks during the Deformation of Zinc Bicrystals at Elevated Temperatures

Zinc bicrystals with a high-angle tilt grain-boundary of $2\theta = 84°$ (fig. 1) and containing a $[11\bar{2}0]$ slip direction common to both crystals in the plane of the boundary, were grown from the melt in two grades of zinc containing 100 ppm and 1 ppm impurities (spectrographic analyses are presented in table I). Specimens of approximate dimensions $2 \times 0.65 \times 0.25$ cm were obtained from the as-grown bicrystals by chemical sawing and electropolishing to ensure deformation-free surfaces. The specimens were loaded in shear in the sense shown in fig. 1, using a gripping arrangement which resulted in slip on basal planes intersecting both the grain-boundary and the grips. Tests were performed in an atmosphere of high-purity argon over the temperature range 20 to 175° C at a constant rate of grip displacement of 300 μm/h.

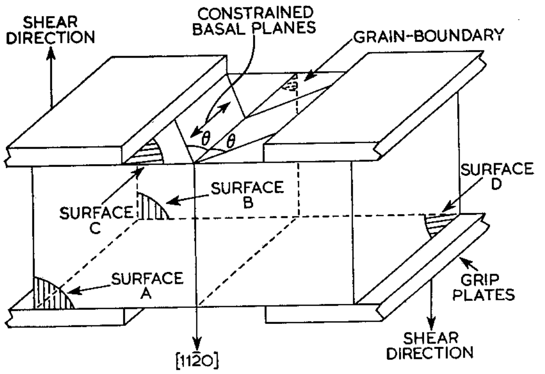

Figure 1 The bicrystal orientation, nomenclature and loading configuration.

TABLE I Spectrographic analyses of zinc bicrystals (in ppm).

	Ag	Cd	Cu	Fe	Pb
4–9's zinc	1	5	10	5	10
6–9's zinc	—	0.1	0.1	0.1	0.1

The deformation behaviour of the bicrystals over the temperature range 20 to 175° C is presented in fig. 2 in terms of the shear stress on the grain-boundary versus grip displacement. The discontinuous changes in the curves are attributable to the formation of kink bands in those parts of the component crystals which were in contact with the edges of the grips.

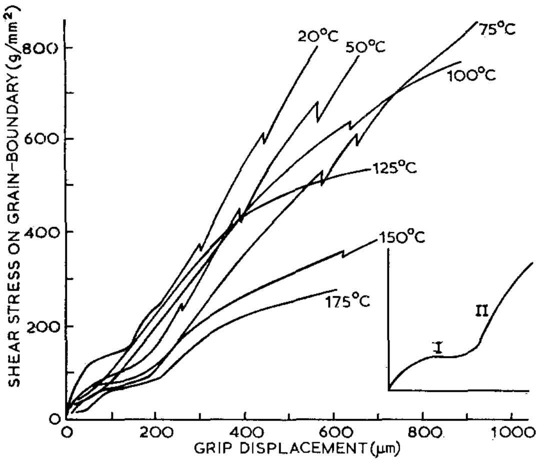

Figure 2 Deformation curves for bicrystals of 6-9's purity zinc with $2\theta = 84°$ tested over the temperature range 20 to 175° C.

Optical examination of the deformed bicrystals revealed that below 75° C the grain-boundaries were intersected by many fine slip bands. In specimens tested at 100 and 125° C grain-boundary cracks were observed on surfaces C and D of the bicrystals (fig. 1). The cracks were associated with those parts of the grain-boundary which were intersected by coarse basal slip bands from one of the component grains (fig. 3). When slip was continuous across the boundary, that is, slip bands from both grains intersected at the same point on the grain-boundary, no cracks were observed. Removal of surfaces layers from bicrystals exhibiting grain-boundary surface cracks revealed the presence of rows of small cavities which are thought to delineate the grain-boundary position before and after migration (fig. 4). The presence of grain-boundary cracks in a specimen in which grain-boundary migration occurred during deformation at 125° C is shown in fig. 5. It can also be seen that the fiducial marks on the specimen surface are slightly displaced, indicating that grain-boundary sliding occurred during test. In bicrystals subjected to grip displacements of ~ 800 μm at 100 and 125° C, the extent of grain-boundary sliding, as measured from the displacements of fiducial marks in the planes of the surfaces, was 1 to 2 μm on surfaces C and D and 10 to 20 μm on surface A. No sliding was detected on surface B.

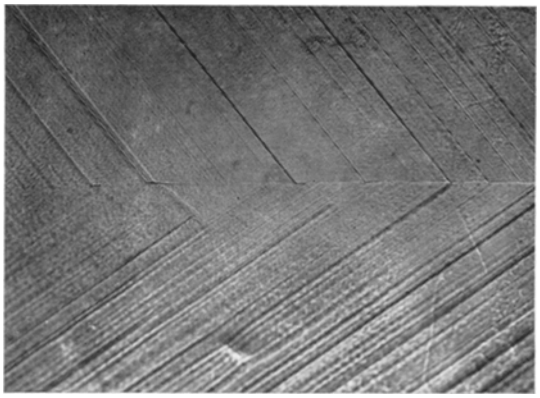

Figure 3 Grain-boundary cracks associated with slip bands intersecting the grain-boundary on surface C after test at 100° C (×162).

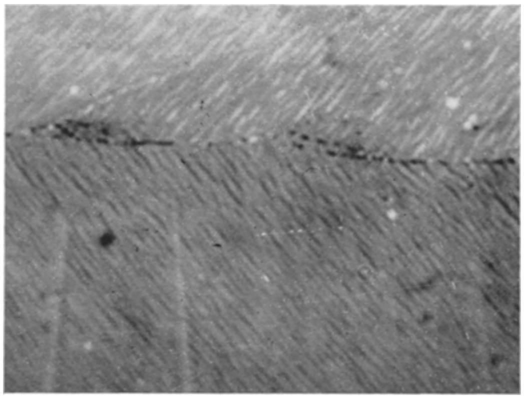

Figure 4 Small cavities observed on an internal section parallel to surfaces C and D after removal of a surface layer. The specimen contained grain-boundary surface cracks and was tested at 100° C (×2800).

Figure 5 Bicrystal tested at 125° C showing grain-boundary cracking on surface C and subsequent grain-boundary migration (×440).

At temperatures above 125° C, whilst coarse slip bands intersected the grain-boundaries, extensive migration occurred and no grain-boundary cracks were observed. Thompson and Bell [1] found that in tests over the temperature range 20 to 200° C on 4-9's polycrystalline zinc subjected to strain rates of 1.0 and 0.1 %/h, a minimum in ductility occurred over approximately the same temperature range, 100 to 125° C, as that for grain-boundary cracking in the bicrystals.

Interrupted tests were carried out at 100° C to investigate the inter-relationship between grain-boundary crack formation and the deformation characteristics of the bicrystals. No grain-boundary cracks or cavities could be detected during stage I deformation (inset to fig. 2) but crack formation was observed in the early stages of stage II, and the number of cracks observed over a limited portion of the boundary increased with overall deformation, as is shown in fig. 6. This observation is consistent with the conclusions of Price [2,3] and Greenwood [4], namely, that cavity nucleation occurs continuously during creep deformation.

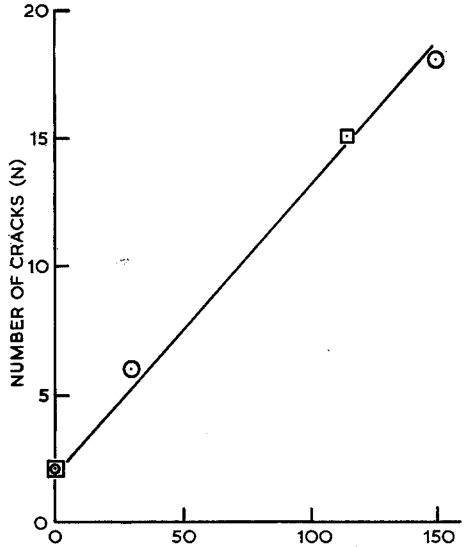

Figure 6 The increase in the number of cracks (*N*) on surface C with grip displacement after the onset of stage II deformation in bicrystals of 4-9's purity zinc, ($2\theta = 84°$), tested at 100° C. The squares and circles represent results obtained from tests on two separate bicrystals.

Single crystals of zinc which deform by basal glide can exhibit extensive regions of low work-hardening [5] (stage I) and the severe limitation of this region in the present tests (fig. 2), leading to the early onset of stage II deformation, can be associated with the intersection of basal slip with the grips and grain-boundary. Surface grain-boundary cracks were only formed during stage II deformation in the temperature range 75 to 125° C if the grain-boundary was intersected by slip bands formed in one of the component grains, and if the intersected region of the grain-boundary did not migrate. These results suggest that crack nucleation was a result of the stress concentrations generated by the interaction of dislocations on basal planes with the grain-boundary.

Smith and Barnby [6] have modified Stroh's [7] original analysis and have shown that the condition for the nucleation of a crack by a pile-up of dislocations of one sign can be expressed as in equation 1:

$$\sigma_E = \left[\frac{\pi G \gamma}{2(1-\nu)L}\right]^{\frac{1}{2}} \tag{1}$$

In this expression σ_E is the shear stress on the glide plane of length L, G is the shear modulus, and γ is the surface energy term involved in crack nucleation. The shear stresses supported by the grain-boundary in stage II deformation were in the range 150 to 300 g/mm^2 and grain-boundary crack formation occurred over this stress range. The shear stresses on the glide planes would also be of similar magnitude, and substituting these values together with the glide plane length of 0.22 cm and $G = 3.5 \times 10^{11}$ dyn/cm^2 in equation 1, values of γ of $\sim$ 60 and $\sim$ 240 erg/cm^2 are obtained. These values of γ appear to be rather low compared with the generally accepted value of γ of $\sim 10^3$ erg/cm^2 for grain-boundary cracking [8]. On the other hand, whilst the present tests were performed in a high purity argon atmosphere, there may have been some surface impurity absorption which could have led to a reduction in the observed value of γ.

When the grain-boundary was intersected by two slip bands at the same point on the grain-boundary, no surface cracks were observed. The movement of dislocations in slip bands in the two component crystals must have developed displacements in the opposite sense to accommodate the shear imposed by the movement of the grips. Thus, the dislocations which would contribute to pile-ups on either side of the grain-boundary would be of opposite sign and as the bicrystals were symmetrical the stress fields from these pile-ups would tend to be equal and opposite. Under such conditions, the stress concentrations would be significantly less than those arising from the pile-up of dislocations of one sign and crack nucleation would not occur as readily.

The cavities or cracks on internal grain-boundaries, observed when sections were prepared parallel to surfaces C and D, tended to be in rather discrete groups (fig. 4). The size of the individual cracks or cavities was in the range 0.5 to 1.5 μm and the groups of cracks extended over a grain-boundary length of approximately 6 μm. The width of the slip bands on the surfaces of the bicrystals (fig. 3) was in the region of 4 to 6 μm. The fact that no internal cavities were observed until stage II deformation occurred, and the apparent correlation between slip band width and length of grain-boundary preferentially containing cracks, supports a model for internal crack nucleation which directly involves slip intersection with the grain-boundary. It should be noted that the length of the surface cracks was always in excess of the slip band width and their formation, therefore, is unlikely to be entirely due to a process of interconnection of smaller cracks formed within the slip band region.

Models [9 - 11] that have been proposed for grain-boundary cavity nucleation try to satisfy two conditions; one, the generation of a sufficiently high stress for decohesion, and secondly, a means whereby the initial nuclei can grow to a stable size. For cavities nucleated at second-phase particles or ledges on grain-boundaries, it has been suggested that the growth to a stable size can be achieved by grain-boundary sliding, provided that the rate of sliding opens the cavity faster than the rate at which sintering can occur. Harris [11] has pointed out that cavity nucleation at a ledge or small step on a grain-boundary as suggested by Gifkins [9] and McLean [10] may require extremely high sliding rates of the order of 10^2 cm/h. The average sliding rates of grain-boundaries on surfaces C and D were $\sim$ 0.4 to 0.7 μm/h. As both surface and internal cracks were formed at these relatively low sliding rates, it is suggested that grain-boundary cracks can grow as a result of the "feed-in" of dislocations from the intersecting slip bands [12]. In this situation the role of grain-boundary sliding would not be as critical as in the model suggested by Harris [11] but the sliding could still contribute to the growth of the cavity.

It has been demonstrated that grain-boundary precipitates can play an important role in the nucleation of cavities [11]. In the present work, no precipitates were evident on the grain-boundaries in either of the two purities of zinc (table I) in which grain-boundary cracks were formed. However, sub-microscopic precipitates may have been present and there is always the possibility of slip band-precipitate intersections leading to grain-boundary crack formation. The presence or absence of grain-boundary precipitates, however, does not detract from the general experimental observations of this note, which support and are consistent with a model for grain-boundary cracking which involves the intersection of grain-boundaries by slip bands. It is not proposed that this is applicable to all materials under all test conditions, but alternatively it should not be discounted as a mechanism for grain-boundary cracking in materials with limited slip systems, such as close-packed hexagonal metals.

Acknowledgements

The authors are indebted to Professor G. V. Raynor FRS, for the provision of laboratory facilities and to UKAEA, Harwell for financial support.

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5 May 1968, and in revised form 2 August

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