C-14 Plastic Flow and Crack Nucleation in Magnesium Oxide

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Abstract

Experiments are described which suggest that fracture nucleation in magnesium oxide in the presence of plastic flow may depend on two factors, (i) the dynamic situation of a dislocation band intersection and (ii) the stress concentrating effect of an impurity precipitate. The fact that on this view crack nucleation would depend upon the chance coincidence of these two factors goes some way to explaining the observed irreproducibility in the occurrence of fracture.

1. Introduction

It is well established that cracks can be nucleated in magnesium oxide by plastic flow (for example(1)). However, the mechanism of crack nucleation is not at all clear; the dislocation band geometry associated with the crack nucleus is frequently consistent with the mechanisms associated with Stroh(2) or Cottrell(3) but this is no proof that these mechanisms are responsible(4). One observational feature that must be accounted for in any comprehensive explanation is the relative rarity of crack nucleation, and the fact that dislocation configurations apparently identical to those that can produce cracking do not always do so(5). More recently Argon and Orowan have suggested a macroscopic mechanism of crack nucleation(6); the mechanism is macroscopic as opposed to the microscopic dislocation theories of Stroh and Cottrell, in that it involves the build-up of macroscopic internal stresses in the crystal. An an example of this mechanism consider the case illustrated by Figure 1, which also represents the principle experiment described in this paper. When two simultaneously active dislocation bands, A and B, broaden and meet, they are unable fully to inter-penetrate at room temperature, and a tilt boundary XY forms along the plane of intersection. Argon and Orowan suggest that the internal stresses associated with this boundary may reach the theoretical strength of the crystal and so cause crack nucleation. They emphasise the macroscopic nature of the process which they clearly distinguish from previous theories of crack nucleation caused by 'stresses due to a few dislocations or a pile-up of dislocations'. The end of the boundary at which the crack should be nucleated depends on the sense of the tilt boundary which in turn depends on whether the experiment is carried out in compression or in tension.

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In a previous paper(5) it has been suggested that such high stresses may not be able to develop because they are continually relieved by the operation of an efficient strain accommodating entity, the mobile bend zone. Bend zones form and move in such a way as to accommodate, at least partially, the strain due to the tilt boundary. The authors presented evidence for linking the crack nucleation event with the dynamic situation of a dislocation band intersection rather than with the tilt boundary stresses. However, these stresses cannot completely be accommodated by the mobile bend zones and it was suggested that once the crack was nucleated its subsequent growth could still be dominated by the macroscopic stresses discussed by Argon and Orowan.

There is an experiment that should help to distinguish between these viewpoints. Consider again Figure 1(c). According to Argon and Orowan (see their Figure 10, reference 6) the macroscopic stress field of the tilt boundary XY should cause crack formation at X or Y, depending on whether the applied stress is tensile or compressive, respectively. In both previous papers(5,6) the crack was reported to form at Y in compression, and Briggs et al(5) showed also that it formed in the dynamic situation of dislocation band intersection. In this case the position of cracking was therefore consistent with both views. Argon and Orowan further suggested (reference 6 page 1034 and Figure 10) that if the experiment were carried out in tension, then the crack must develop at X, after a critical length of tilt boundary had formed. However, the dynamic situation would still be at Y, so in the view of Briggs et al. crack nucleation, if it occurred, should be at Y also. Hence, we have an experiment which should help to decide which of the two phenomena - the build-up of the macroscopic strain field due to the tilt boundary, or the dynamic situation at a dislocation band intersection - is the more important to crack nucleation. This is the principal experiment to be described in the present paper.

2. Experimental

Specimens with reduced gauge sections were prepared initially free from 'fresh' dislocations, and dislocation sources were then introduced in a controlled manner by the techniques described previously(5). The dislocation band intersection experiments to be described were performed at constant stress on the tilting beam machine(5). All tests were observed in transmitted plane polarised light, and were recorded on Eastman Kodak Negative colour film. Subsequent examination and photomicrography was carried out on a Zeiss Ultraphot II microscope. Dislocations were revealed by etching in a mixture containing equal parts of saturated ammonium chloride solution and concentrated sulphuric acid, and impurity precipitates by etching in fuming nitric acid(7), or by ultramicroscopy(8).

3. Results and Discussion

The critical experiment referred to in section 1 was performed seven times. In six of the seven cases cracks did not occur near the tilt boundary although the latter developed to lengths of 0.2 (twice), 0.25, 0.7 and 1.5 mm in 1.5 mm wide specimens, and 1.5 mm in a 1.7 mm wide specimen. Fracture occurred elsewhere in the crystal in the first four cases, and not at all in the last two. In the remaining case a crack was nucleated at Y when the tilt boundary was almost 1.5 mm long, after one of the dislocation bands had stopped widening. The development of the tilt boundary in this case is shown in the extracts from a cine film presented in Figure 2. In the line diagram of Figure 2, which corresponds to the last cine film extract, are shown the bend zones that limit the macroscopic stress induced by the tilt boundary XY. The principle stress difference, calculated from measurements of the birefringence made after removal of the load, at the point Y, did not in six of these cases exceed 3 x 10^9 dyn.cm^{-2}, and did not vary significantly with the length of the tilt boundary. Since fracture occurred at Y in the remaining case, no measurement was possible.

These experiments show that:

(i) Crack nucleation that is associated with tilt boundaries is not a common occurrence in carefully prepared crystals. This fact strengthens the view that the mobile bend zones efficiently accommodate the tilt boundary strain.

(ii) When crack nucleation does take place, it still occurs in the position where moving dislocations are meeting, even when the macroscopic stresses favour crack nucleation at the opposite end of the tilt boundary.

Argon and Orowan discuss a possible example of crack nucleation in the position to be expected on their hypothesis (reference 6 page 1034, the experiment described by Parker). However, this example is the same as that discussed by Washburn et al.(10), and the crack is probably nucleated along the line of intersection of dislocation bands; alternatively it may be an example of the dislocation-assisted growth of a pre-existing crack (for discussion of this see reference 11).

Thus while these experiments place emphasis on the dynamic nature of crack nucleation (this was also commented on by Stokes et al., reference 1) they still leave open the problem of why apparently identical dislocation configurations produce variable results in respect of crack nucleation. Briggs et al(5) suggested that the heterogeneous distribution of impurity precipitates might provide the reason for the experimental variations. It is known that the larger impurity precipitates act as fracture origins in magnesium oxide in which plastic flow has been suppressed by neutron irradiation(12). Applied stresses of ~ 10^{10} dyn.cm^{-2} are needed to cause these precipitates to act as crack nuclei; such stresses might momentarily be reached in the dynamic situation of a dislocation band intersection, especially in the presence of the dislocation avalanches that occur there(5). In this case the irreproducibility of the fracture phenomena would stem from the random nature of both the precipitate distribution and the avalanche occurrence.

As one check of this suggestion the impurity distribution around the fracture origin was studied. The actual fracture origin can readily be identified by the associated microstructure and there is an etchant that produces pyramids on impurity precipitates(7). However, this is not an easy experiment to carry out because of the difficulty in producing crack nucleation in a controlled manner; furthermore, to be of any value the fracture origin has to be identifiable within a very small area because the impurity precipitate density is often ~10^7 cm^{-2}(7). The necessary conditions were obtained in two experiments in which a tilt boundary was grown almost parallel to the tensile axis. The dislocation band configuration is shown in Figure 3(a). One dislocation band EF became established before it was intersected by a second band. After intersection the bands widened simultaneously to form the tilt boundary shown in Figure 3(b). Fracture occurred from the line of intersection at G, and spread across a plane perpendicular to the tensile axis. In both experiments in which fracture occurred in this manner the fracture origin was clearly identifiable. One fracture face is shown in Figure 4(a), and the fracture origin is arrowed. The fracture origin lay on the trace CD of the tilt boundary on the fracture face. In each of the two cases etching in fuming nitric acid produced a pyramid exactly on this fracture origin (Figure 4(b)), thereby showing the presence of a precipitate. Furthermore, in both cases, etching also produced a pyramid at the fracture origin on the matching fracture face; this shows that the fracture also intersected the precipitate.

These two observations do not prove that all fracture origins have precipitates, but are at least consistent with the view that crack nucleation may be associated with an impurity precipitate.

4. Conclusions

The experiments reported in this paper together with previous work suggest that:

(a) A tilt boundary by itself does not generally cause crack nucleation because the strains it induces are partially accommodated by mobile bend zones. The importance of tilt boundaries lies in their determining the geometry of crack growth rather than crack nucleation.

(b) The dynamic situation existing momentarily at a dislocation band intersection, and an impurity precipitate, may play a joint role in crack nucleation. The random occurrence of these features may explain the unpredictable nature of crack nucleation.

References

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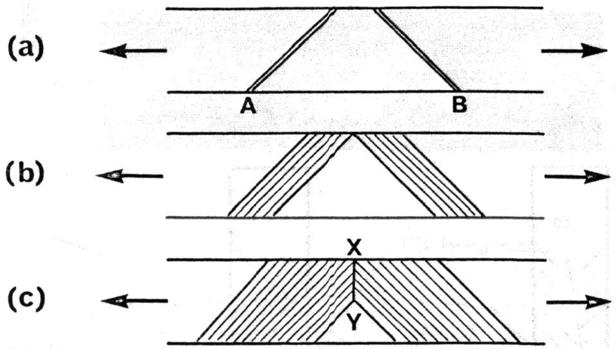

Figure 1

A schematic diagram showing the intersection of two dislocation bands (A and B) to form a tilt boundary XY. This corresponds with the film sequence in Figure 2.

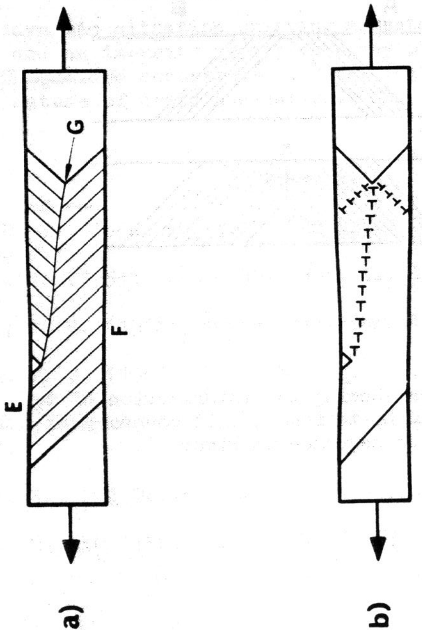

Figure 3

Schematic diagrams showing:

(a) the dislocation band geometry, and
(b) the tilt boundary and bend zones formed before fracture occurred along the line of intersection at G.

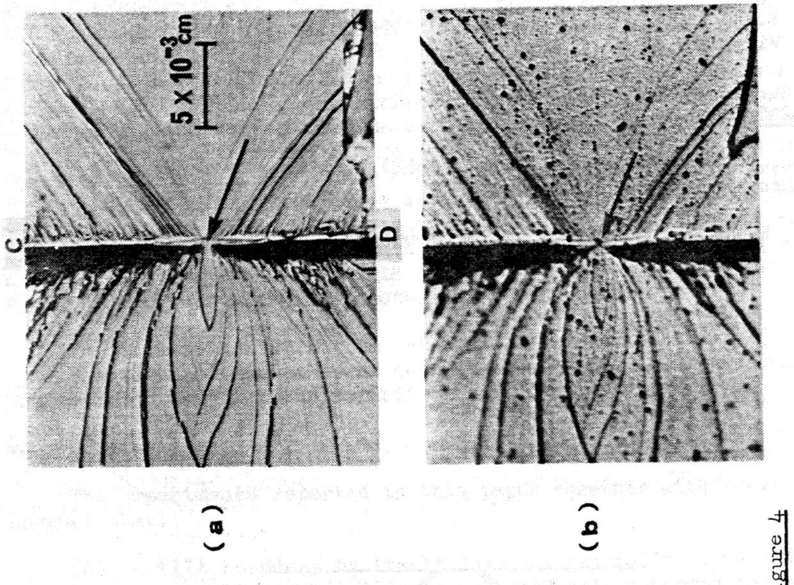

Figure 4

Photomicrographs showing:

(a) the fracture origin (arrowed) lying on CD, the trace on the fracture face of the tilt boundary depicted in Figure 3, and
(b) the black pyramid (arrowed) produced on the fracture origin by etching in fuming nitric acid.

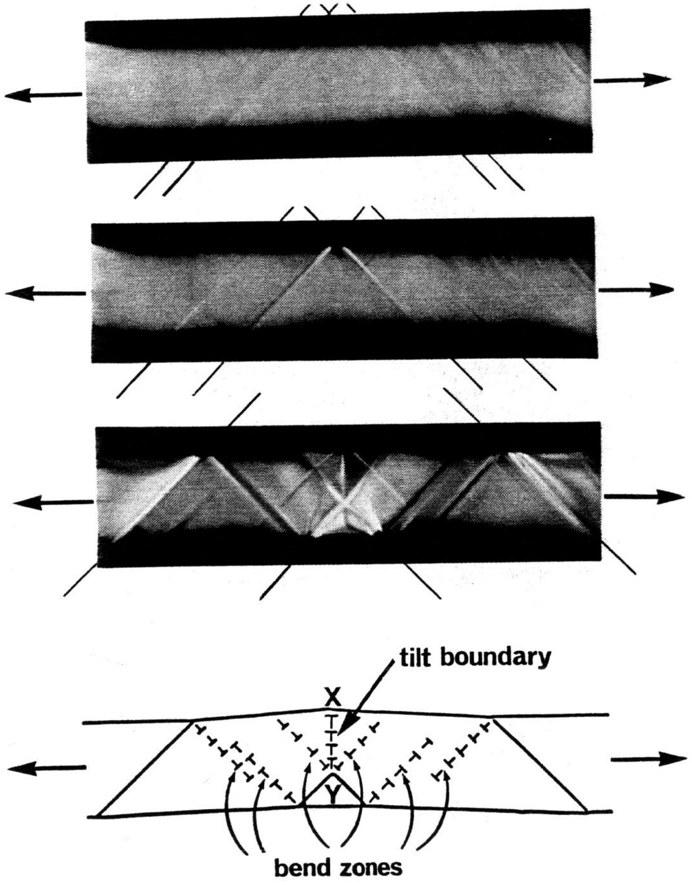

Figure 2 Extracts from a cine-film showing the development and intersection of two dislocation bands, and a schematic diagram showing the tilt boundary and bend zones that formed.