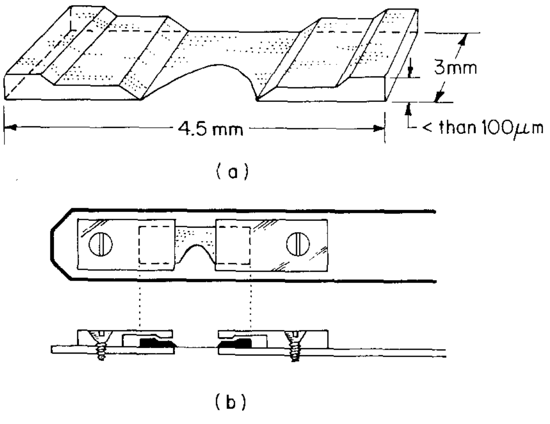
Fracture in magnesium oxide bicrystals

Since Zener [1] first pointed out that blocked dislocations would cause stress concentrations that might lead to fracture, several models for the nucleation of a crack have been proposed by Mott [2], by Orowan [3], by Stroh [4], by Cottrell [5], and by Gillman [6].

From bending experiments with magnesium oxide bicrystals, Johnston *et al.* [7] Clarke *et al.* [8], and Ku *et al.* [9] have shown that a crack can nucleate at a grain boundary in magnesium oxide. From the metallographic study on compressed bicrystals of magnesium oxide, Westwood [10] has shown that a crack can be formed at a boundary by piled up edge dislocations.

In the present study the mechanism of fracture in magnesium oxide bicrystals was investigated by direct continuous observation of dislocation motion and fracture initiation during extension of a thin foil of a magnesium oxide bicrystal. The bicrystals used were purchased from the Norton Company, and these crystals typically contain about 500 ppm impurities. Thin sheets approximately 1 mm thick were cut with a string saw, carefully polished on silicon carbide paper to about 0.3 mm thickness and them chemically polished in hot H_3PO_4 solution to remove the surface microcracks. The final thickness was about 0.1 mm. Thin foils of magnesium oxide bicrystal samples were then prepared by directing a jet of H_3PO_4 at the area to be thinned [11]. The thin foil specimen was extended by a tensile device in a 100 kV electron microscope as shown in Fig. 1. In order to prevent pieces of a fractured specimen from falling into the microscope, a carbon coating was applied to one side of the sample. The average extension velocity was 0.1 μm sec^{-1}, and to analyse dislocation motion the deformation/fracture process was recorded on video tape; subsequently, images were filmed with a 16 mm camera at a speed of 24 frames per second.



(a)

(b)

Figure 1 (a) Sample configuration and (b) attachment to the extension of the device.

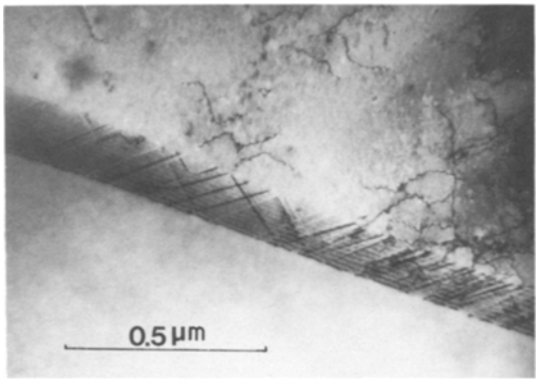


Figure 2 A typical grain boundary in a magnesium oxide bicrystal.

Dislocation features at the grain boundary of the magnesium oxide bicrystal foil are shown in Fig. 2. No impurity precipitates were observed at the grain boundary, and the edge dislocations initially present at the boundary did not move under extension. Fracture in these samples always occurred not at the grain boundary, but in the bulk crystal adjacent to a boundary. Even after fracture, the original edge dislocations remained in the same position as before extension. This is thought to result from the edge dislocations being pinned by impurities and, therefore, rendered immobile. The fact that not only edge dislocations at grain boundaries but also grown-in dislocations do not move under extension indicates that the pinning effect of impurities on dislocation motion is extremely strong.

Slip lines are formed by screw dislocations parallel to the extension axis moving on {1 1 0} planes at right angles to the axis. The moving screw dislocations usually leave a trail of edge dislocations behind. The subsequent motion of these edge dislocations appears to create slip lines at a right angle to the extension axis. The slip lines increase in number during extension as illustrated in Fig. 3. The density of slip lines changes

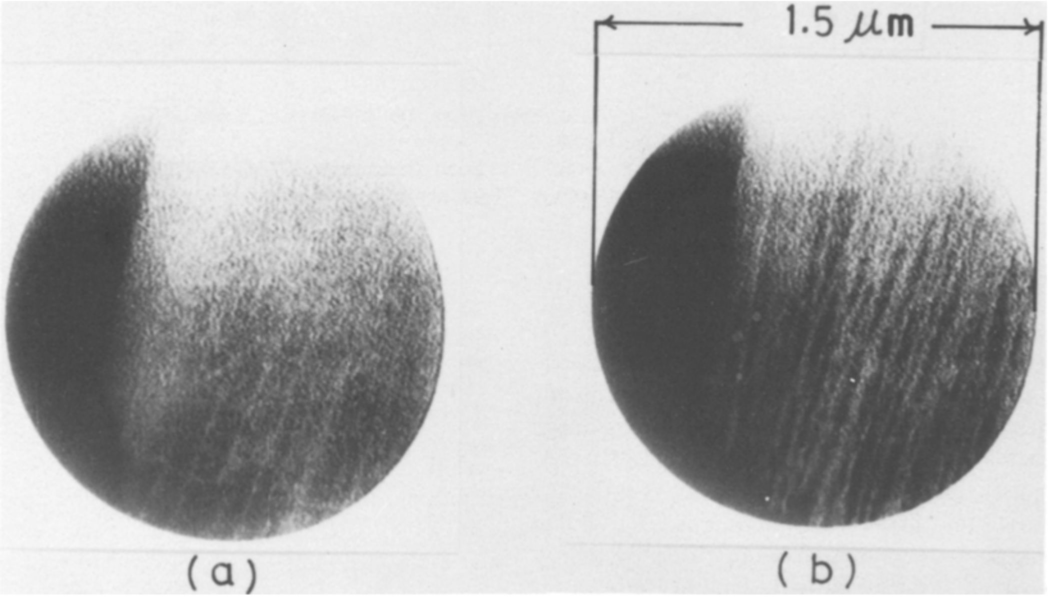


Figure 3 Change of slip line density (a) before extension and (b) at the final stage just prior to the fracture.

from about $9 \times 10^4\,\text{cm}^{-2}$ at the initial stage, to $2.3 \times 10^6\,\text{cm}^{-2}$ at the final stage just before fracture. Fig. 4 shows the part of a foil immediately adjacent to the fracture line. It is clear that the slip line density near the grain boundary is lower than in the bulk adjacent to the boundary. Direct evidence for boundary segregation is found in autoradiographic or X-ray emission photographs for impurities in furnace-cooled magnesium oxide bicrystals [12, 13]. In our specimens, assuming that impurity solutes are more concentrated in a region adjacent to the boundary in which dislocation motion is hindered, it is reasonable that slip line density was low in this region as compared with the bulk. The width of the low slip line density (grain-boundary width) was measured to be about 540 Å at one side of the boundary and 2200 Å at the other side. The difference in grain-boundary width between the two sides of the grain boundary may indicate that the amount of impurity solute depends strongly on the relative orientation of adjacent grains.

The fracture mainly occurs along (1 0 0) planes with some steps on (0 1 0) planes as shown in Figs. 5 and 6, but sometimes fracture was observed along the (1 1 0) plane. In evaluating the process of crack formation in a single crystal, we must

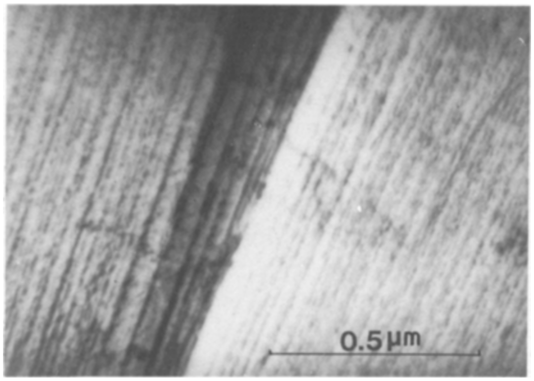


Figure 4 Features of slip lines near a grain boundary after fracture.

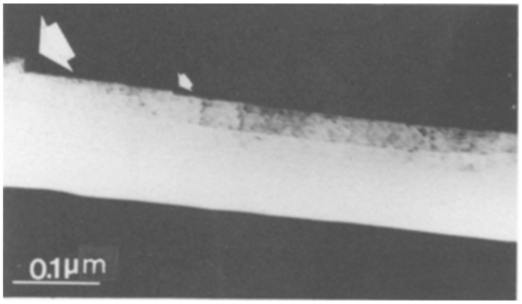


Figure 5 The surface of the fracture. Arrows indicate steps described in text.

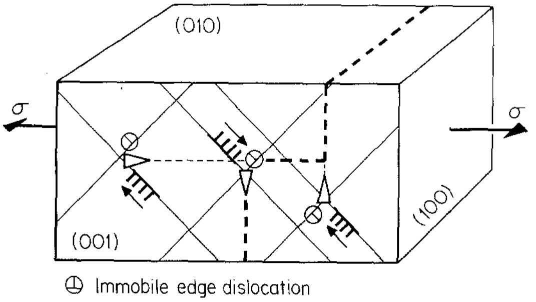


Figure 6 Proposed mechanism of crack formation.

keep in mind two experimental facts found by direct observation. One is that grown-in dislocations do not move; the second is that the slip line density is low near grain boundaries in comparison with the crystal bulk. That is, the pinning effect of impurity solutes on dislocation motion is extremely strong. From these observations it is inferred that crack formation in magnesium oxide occurs by the Stroh mechanism with impurity solutes or grown-in dislocations associated with impurity solutes acting as obstacles on which edge dislocations pile up. If the relation between moving dislocations and immobile grown-in dislocations satisfy the condition of crack formation given by the Cottrell model [5], cleavage fracture can occur under extension (Fig. 6).

In summary, fracture observation in bicrystal magnesium oxide indicates that fracture occurs not along the grain boundary, but in the bulk about 500 to 2000 Å from the boundary. This observation suggests a serious caveat associated with the interpretation of the chemical composition of so-called "grain-boundary" fractures, since these fractures may often be adjacent to, rather than at, the grain boundary.

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High pressure X-ray studies of polymers. I. Calibration for the diamond-anvil cell in the range 0 to 15 kbar

The diamond-anvil pressure cell, first reported in 1959 [1], has been used to study materials at high pressures with a variety of methods, including optical observation [2], X-ray diffraction [2–8], absorption spectroscopy [9], and Raman scattering [10]. The most serious drawback in using the diamond-anvil pressure cell is the measurement of the high pressure attained in the device. The usual method is to incorporate an NaCl standard with the sample and to make measurements of the lattice parameter using X-ray diffraction. The pressures can be determined through an equation of state [11]. Recently, a more convenient method has been reported by Piermarini and Block