10. Fracture of Ceramic Materials

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ABSTRACT

This chapter describes microscopic observations of deformed and etched MgO single crystals. It states that etch-pit studies reveal slip bands densely populated with dislocations and that these slip bands act as barriers to dislocations on intersecting slip planes. Cleavage cracks on {001} and {011} planes were found at the junctions of intersecting slip bands. The formation of these cracks was evidently associated with the high local stresses produced by dislocation pile-ups.

Introduction

Although some progress has been made toward the understanding of processes whereby a cleavage crack is nucleated in a metal, no direct evidence has as yet been presented that clearly shows the nature of the crack nucleation process. The reasons for this are straightforward. Metals tend to undergo relatively large amounts of plastic flow before breaking; the large number of dislocations surrounding incipient cracks makes it difficult to assess the role played by dislocations in nucleating the crack. Furthermore, etch-pit and other techniques have not been particularly successful in revealing the locations of dislocations in a deformed metal. It is interesting to note, however, that the mechanisms of slip and fracture can be studied in great detail in ionic and covalent crystals.[1–3] Such materials are transparent, and so dislocation structures can be observed directly when decoration techniques are employed.[4] Also, the progress of advancing cracks can easily be followed by light reflection from the fracture surface. Of great importance, however, is the fact that cubic ionic crystals generally slip a small amount before a crack forms. The number of dislocations present in the cracked material is small compared with metals, and furthermore, these dislocations are readily revealed by reliable etch-pit techniques. During the past year, a great deal has been learned about how cracks are nucleated in such materials.[5–8] The object of this chapter is to present a summary and interpretation of the results of this work.

Plastic Deformation

It has been known for a long time that certain ionic solids, particularly those with cubic crystal structures, are ductile under certain conditions.[9,10] More recently, it has been shown that even the refractory oxide MgO is ductile at room temperature when tested in single-crystal form.[11] When such crystals are loaded along a [001] direction, they deform elastically up to a rather well-defined stress and then yield to flow plastically without the occurrence of much work hardening. A typical stress-strain curve is shown in Fig. 1. Flow occurs on the four systems [101] $(\bar{1}01)$, $[\bar{1}01]$

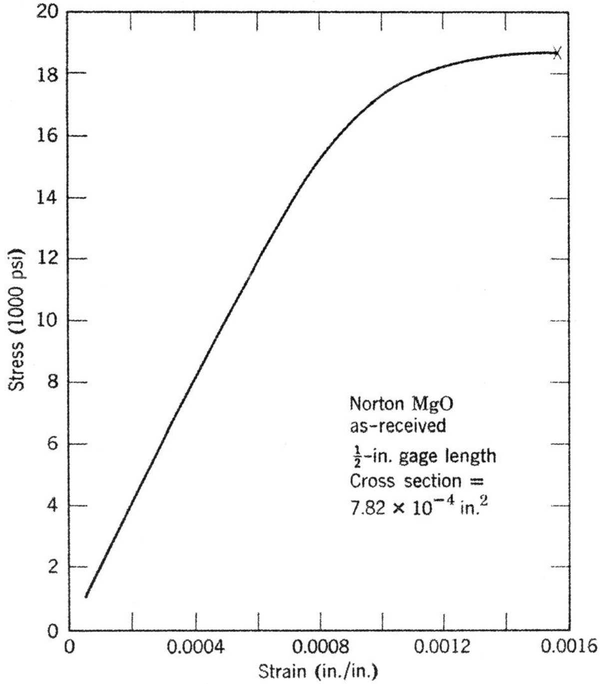

Fig. 1. Stress-strain curve for MgO single crystal loaded in tension.

(101), [011] $(0\bar{1}1)$, and $[0\bar{1}1]$ (011). Dislocations seem to originate mainly at surfaces, but in random location. Some loops start at obvious surface defects, such as cleavage steps or surface cracks. Others, however, appear where no visible defects exist. Dislocations from the first few sources activated move completely across the specimen, leaving a wake of secondary loops on nearby parallel planes. The point where each loop intersects the external surface can be revealed by etching with a solution containing 10% NH_4Cl and 30% HCl,[6] or with a solution consisting of five parts saturated NH_4Cl, one part concentrated H_2SO_4, and one part water.[8] Observations of expanding loops have shown that in MgO the edge components move faster than the screw portions,[6] thus forming elliptical loops. The first few dislocations in a slip band move long distances; the secondary loops, however, generated as they are in nearby parallel planes because of the high local stresses associated with the primary dislocations, often expand less because their mutual interactions cause them to become stuck in the lattice.

Measurements made in one experiment showed that strains of 10% occurred within a slip band. This value of strain could be produced by the passage of one $a/2\langle 110\rangle\{1\bar{1}0\}$ dislocation across every twentieth layer of atoms. However, etch-pit studies showed that about 10^{10} dislocations per square centimeter remained in the slip band. An appreciable amount of the total strain could be accounted for on the basis of the dislocation loops that had expanded over small areas. For example, 10% strain could have been produced by 10^{10} dislocations per square centimeter, with each dislocation moving through a distance of about five microns. To obtain a clearer insight into the nature of a slip band, some deformed crystals were annealed. This treatment permitted small loops to collapse or neighboring pairs of dislocations of opposite sign to unite and thus reduce the density of dislocations in a slip band. Re-etching after annealing showed clearly that only about 10^6 dislocations per square centimeter remained.

Thus it is evident that the structure of a slip band is complex and that a multitude of local stress fluctuations exist within the band. It is not surprising, therefore, to find that a slip band formed during an early stage of the flow process becomes a rather effective barrier to dislocations induced to move at a later time period on an intersecting slip plane. An example of this is shown in Fig. 2. Studies of slip-band structure have revealed that many intersecting slip bands fail to penetrate the barriers imposed by the regions of initial slip. However, it has been found that in almost all cases slip bands easily penetrated the grown-in substructure boundaries. Such boundaries are rarely effective barriers to slip.

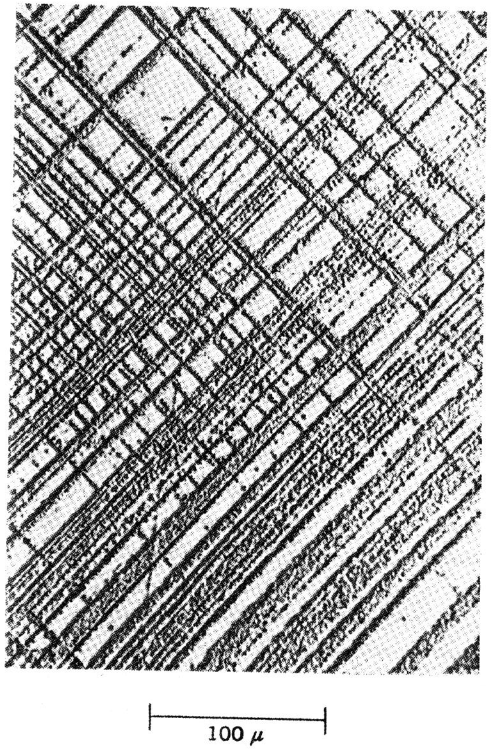

Fig. 2. Photomicrograph showing how slip bands act as barriers to dislocations moving on intersecting planes.

Cleavage Fractures

The condition that leads to the initiation of cleavage fracture in single crystals of materials like MgO is the presence of dislocation pile-ups at intersections of slip bands. This is illustrated schematically in Fig. 3, and actual examples are shown in photographs in Fig. 4. Figures 4*a* and *b* show views of two adjacent crystal faces; the dark vertical lines are the edges of the samples that were parallel to the loading axis. Note that in these cases, the cracks initiated along the lines where the secondary bands impinged upon the primary band. The density of etch pits indicates that the local stress concentrations might easily have become 100:1 or 1000:1 prior to fracture. Since the specimens broke with an average stress of about 10^4 psi, the local stress might thus have reached the theoretical fracture strength of approximately 10^6 psi. Figures 4*d* and *e* show views of only one face on each crystal. In these cases, the

load was applied horizontally with respect to the photographs, and cracks can be seen developing in the top of the pictures where slip bands are shown to intersect. Although cracks were frequently found to start at the corners, they were also initiated in other regions of the specimen. Figure 4*c* shows a crack forming near the middle of a crystal face, but it too was caused by the stress concentration produced locally by dislocations piled up on an intersecting slip plane. Again in this case, the load was applied in the horizontal direction.

It has become evident from fracture studies on MgO that cleavage fractures can be initiated by the high local stresses produced by disloca-

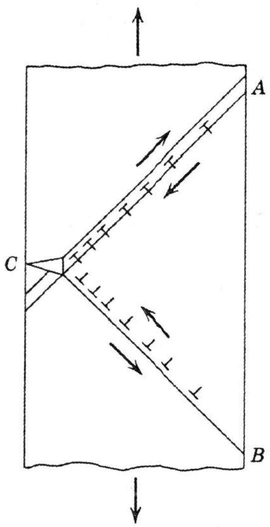

Fig. 3. Sketch showing how dislocation pile-up at intersection of two slip bands can lead to formation of cleavage crack.

tion pile-ups. Since grain boundaries are known to be effective barriers to slip, it is to be expected that in polycrystalline materials cleavage fractures would occur because of dislocation pile-ups at grain boundaries. This has not as yet been demonstrated experimentally, but it is almost certain to be true. It may be concluded, therefore, that if significant amounts of ductility are to be obtained in polycrystalline materials such as MgO, grain size will play an extremely important role. Ductility should only be possible with polycrystalline specimens in which grain size is so small that only a few dislocations can function, and hence pile up, on a single plane. It appears evident that grain sizes in the micron range would be essential for ductility in polycrystalline magnesium oxide. Experiments are now under way to check this hypothesis, but the results will not be available for some time.

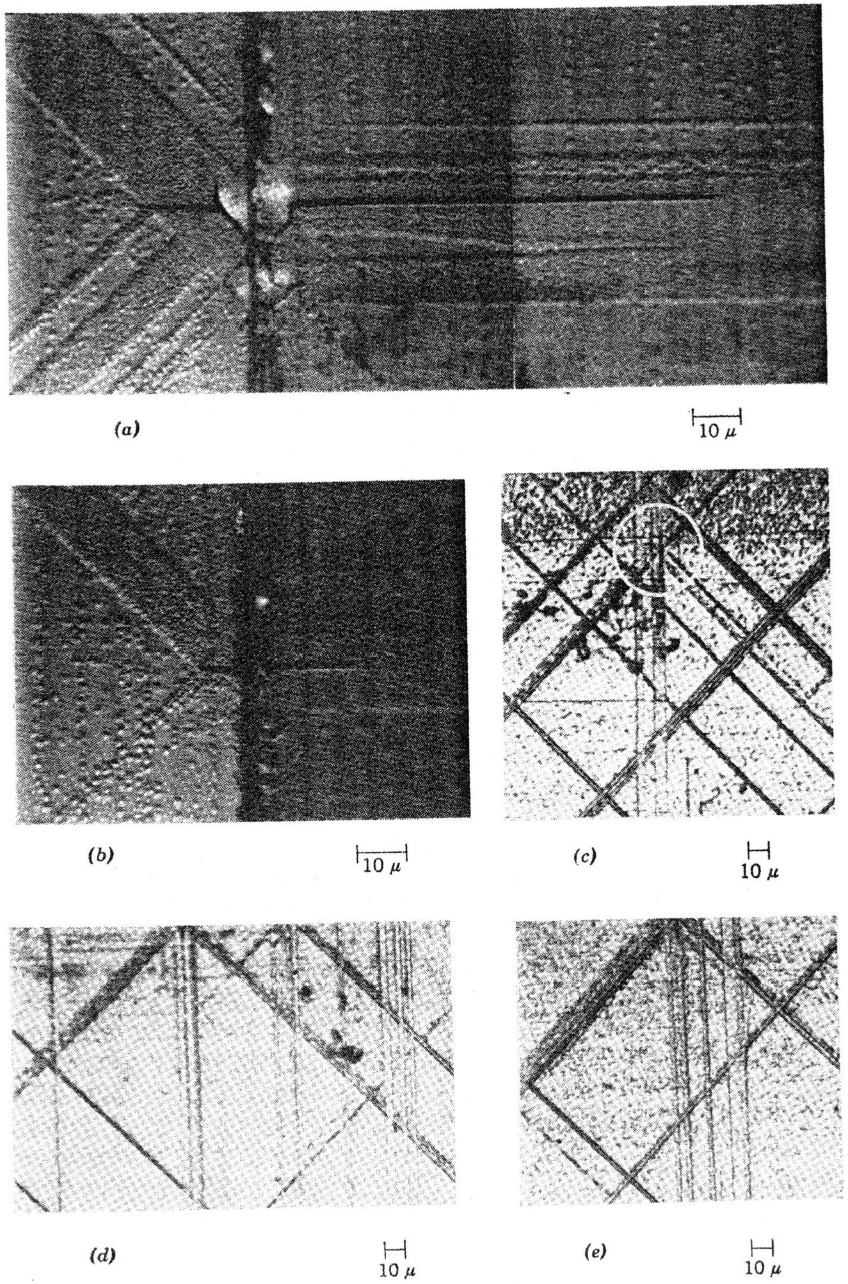

Fig. 4. Typical examples of crack nucleation as observed in MgO.

Initiation of Fracture on Slip Planes

Experiments made on MgO by Stokes, Johnston, and Li [8] have revealed a mechanism responsible for the initiation of cracks forming parallel to slip planes. These investigators tested single-crystal specimens in compression and found that at about 3% plastic strain minute cracks formed at the junctions of intersecting slip bands. These cracks are shown in Fig. 5; they were opened or enlarged by etching after the deformation.

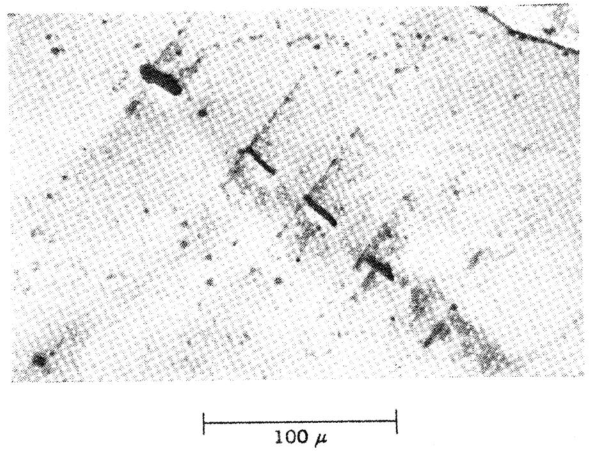

Fig. 5. Cracks formed by dislocation pile-ups in single crystals of MgO loaded in compression. (After Stokes, Johnston, and Li.[7])

The density of dislocations in this specimen was so great that details of the dislocation distribution could not be revealed accurately by etching. However, the cracks shown in this figure were clearly located at the intersections of primary and secondary slip bands. A polarized light photograph of the same region of the crystal was taken before the crystal was etched, and this picture is reproduced in Fig. 6. It indicated that slip on two intersecting sets of planes had occurred in this specimen. The cracks ran parallel to the primary or heavy band, and they originated at the points where secondary bands intersected the primary band.* In regard to the growth, Stokes, Johnston, and Li showed that tiny cracks of the kind shown in Fig. 5 spread rapidly along the length of edge dis-

* The investigators interpreted these photographs somewhat differently. They considered the band parallel to the direction of the cracks to be a kink band. It seems more reasonable to the author to consider this a region of primary slip. The important fact, however, is that this work demonstrates clearly one way in which cracks can form and grow parallel to slip planes.

location lines, thereby forming [100] direction slits. They also observed that two sets of mutually perpendicular cracks frequently formed and found that these were often joined. Macroscopic cracks were presumably formed by the growth and subsequent joining of the microcracks.

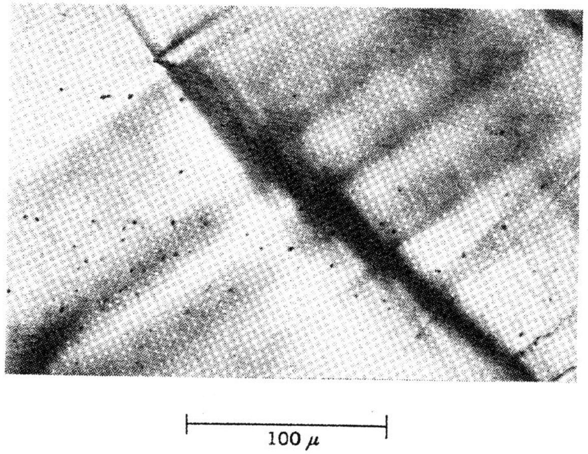

Fig. 6. Same view as shown in Fig. 5 but before etching and as seen with polarized light. (After Stokes, Johnston, and Li.[7])

Discussion

There is no doubt that a physical picture of fracture nucleation in those materials that exhibit even small amounts of plastic flow must be based on a dislocation mechanism. Theoretical and experimental work have shown that many dislocations may accumulate on a single slip plane, particularly when a barrier is present that impedes the motion of dislocations through the lattice. A dislocation pile-up generates a stress concentration, and if the number of dislocations is large, this stress concentration may reach a value of 1000 times the average stress.[12] It was suggested by Zener [13] in 1948 that the stress concentration might become high enough to cause the leading dislocations to coalesce. Such coalescence would produce a crack nucleus as indicated schematically in Fig. 7. After the first two dislocations had united, further coalescence would become easier, and the crack would grow. The opening of such a crack would not necessarily lead to immediate fracture. The crack would extend through the region of high stress concentration at the end of the slip band and would then stop unless its length was greater than the critical value needed for self-propagation. The growth of a crack is clearly dependent upon the hydrostatic component of the stress system,

a component that has little influence upon resistance to plastic flow. Extension of a crack is aided by hydrostatic tension and hindered by hydrostatic pressure.[14]

The concept of the nucleation of cracks by the coalescence of dislocations at the head of a pile-up was developed quantitatively by Stroh.[15] He concluded that the stress would be high enough to cause a crack to form if n dislocations were piled up under a shear stress σ_0 when $nb\sigma_0 = 12\gamma$, where γ is the surface energy and b is the Burgers vector. In deriving this equation, Stroh considered that all of the dislocations piled up on a slip plane contributed to crack nucleation. In a subsequent analysis,[16] however, the mechanism was considered in greater detail, and the view was expressed that a crack is nucleated because of the short-range interactions of a few dislocations at the head of the pile. The remaining dislocations were presumed to produce the high stress concentration required to force the leading dislocations together but not necessarily to unite with the leading dislocations to form the crack. Their action could be replaced by any other suitable stress raiser; for example, the stress concentration produced by groups of dislocations on nearby parallel planes.

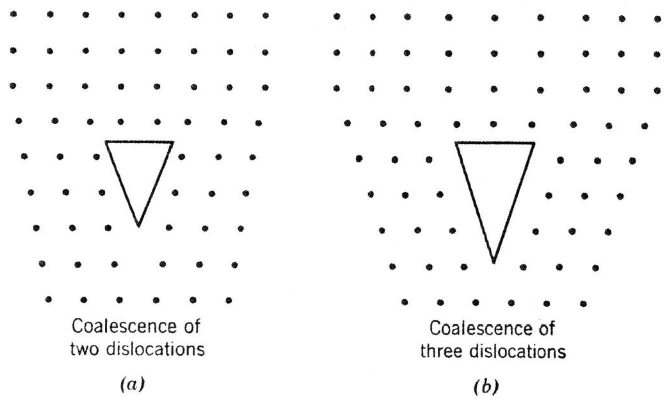

Fig. 7. Sketch showing how dislocations can coalesce to form crack nucleus.

Petch,[17] following the concepts of Stroh, developed a theory to account for the difference between ductile and brittle failure. He also introduced terms that accounted for the temperature dependence of the ductile-brittle transition in the behavior of iron. He suggested that cleavage occurs when the load stresses are high enough to cause a dislocation crack to propagate as a Griffith crack. Ductile fractures presumably occur when dislocation cracks cannot propagate as Griffith cracks because of insufficient elastic strain energy stored in the system. In this case, deformation would continue with the generation and slow growth of many crack nuclei until they eventually became linked together to form a major crack. Petch concluded that the distinction between ductile and cleavage fracture lies in the difference in growth rate rather than in the nucleation mechanism.

The theory of propagation of a Griffith crack in a ductile material involves a term called the "effective surface energy" required to extend the crack. This consists of the true surface energy γ and the plastic work γ'. Plastic work involves the generation and motion of dislocations through the lattice. Consequently, the amount of energy required to extend a crack is related to the number of dislocation sources that operate as the crack advances. In materials where dislocations are generated easily, the plastic work term is large, and Griffith-type crack propagation is difficult. For materials in which dislocations are locked, for example, steel, cleavage fracture becomes a possibility under certain conditions. In materials like MgO and LiF, dislocations are difficult to generate, but once formed they will move through the crystal lattice at stress levels of only a fraction of those needed to induce generation.[8] Crack nuclei in such materials tend to grow as Griffith cracks while they are still small, and therefore, solids of this kind tend to behave in a brittle manner.

Concluding Remarks

The recent experimental work discussed here indicates that two kinds of fractures can occur as a result of the high local stress concentrations produced by dislocation pile-ups. Grown-in substructure boundaries are not effective barriers to moving dislocations unless strongly pinned by foreign atoms, such as carbon in iron. Slip bands that form in crystals during plastic flow are populated by a very large number of small dislocation loops. Such bands can be strongly resistant to penetration by dislocations moving on intersecting slip planes. Microscopic examination of deformed and etched MgO crystals has shown that large numbers of dislocations are present near the points where the secondary bands intersect but do not penetrate the primary band. Etch-pit studies indicate that at these points the number of dislocations is large, and hence the local tensile stress may reach the theoretical fracture strength of approximately 10^6 psi. Small cleavage cracks were found to form on the (001) plane at numerous slip-band intersections.

Another kind of crack has also been observed to form at the intersections of slip bands. Cracks of this kind were observed to run parallel to one of the slip directions. They were evidently nucleated by the coalescence of dislocations at the heads of pile-ups. In materials like ductile metals, where dislocations are easily generated, crack nuclei presumably continue to form with increasing amounts of strain, and these nuclei grow slowly into microcracks that eventually join to form a macroscopic crack.

In some metals, for example, steel, the dislocations may become locked by impurity atoms such as carbon. This may cause the flow stress to increase to the point where cracks may grow by the Griffith process. In materials like MgO, dislocations are generated only at stress levels far above the value of stress required to move dislocations through the lattice. Crack nuclei in materials of this sort tend to grow as Griffith cracks while they are still very small. Hence the fractures tend to be of the brittle type.

In conclusion, it is encouraging to note that theoretical and experimental research on fracture processes has finally reached the stage where an understanding of how and why things break is becoming possible.

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DISCUSSION

R. J. Stokes, T. L. Johnston, and C. H. Li, *Minneapolis Honeywell Research Center.* On the basis of our recent observations, we conclude that the slits in magnesium oxide crystals, described by Parker, are generated by an avalanche of edge dislocations suddenly released along the edge of a secondary slip band. These dislocations pile up against the barrier provided by an orthogonal primary slip band and coalesce to form a crack nucleus. The plane of the slit nucleus is oriented parallel to and lies at the edge of the primary slip band. This microcrack eventually achieves the critical condition for cleavage propagation over the {110} plane and grows to form a microscopic {110} plane slit. The details of this work will be published elsewhere.[1]

At a later stage of its development, the slit switches out of the {110} cleavage plane into the {100} cleavage plane and, in this manner, provides the source for characteristic {100} surface cleavage fracture. Using specimens that have been thoroughly chemically polished (10 minutes in hot orthophosphoric acid) and then carefully handled, we find that cleavage fracture under tension always originates by this mechanism. Such specimens are extremely brittle (generally not exceeding 0.01% elongation) and the ½-in. gage length contains only a few primary slip bands, yet the source of fracture is an internal slit, parallel to the {110} plane and nucleated where two of the slip bands have intersected.

It should be noted that the orientation and location of this source of cleavage fracture is quite different from that described by Parker. The reason for the discrepancy between the two sets of observations is believed to be a matter of surface preparation. Cleaved crystals have been found to contain microcracks along their edge and dislocation loops in their surface, both of which are introduced during the act of cleavage. This surface damage changes the fracture properties markedly; in particular, the source is generally located at the corner of the specimen. It is suggested that the mechanism described by Parker may be the one that gives rise to the corner point source of fracture.

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