JOURNAL OF MATERIALS SCIENCE 17 (1982) 1501–1507

Electron channelling study of fracture in alumina: evidence for crack-tip plasticity

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The fracture surfaces of aluminum oxide specimens broken in bending are examined using selected-area electron channelling. The distorted electron channelling patterns which result are interpreted as possible evidence of crack-tip plasticity. The relationship of these findings to earlier experiments is discussed.

1. Introduction

Whether or not plastic flow is associated with the fracture of aluminum oxide at low homologous temperatures ($\lesssim 900^\circ$ C) has been a subject of investigation for several decades. Arguments for and against plasticity-controlled fracture of sapphire are summarized by Congleton *et al.* [1] and by Wiederhorn *et al.* [2], respectively. The subject is important because it relates to the physical basis of what seemingly should be, but is not [1], an athermal, Griffith-type process, controlled solely by surface free energy.

Studies to date have usually relied upon one or more of three experimental approaches: (1) transmission electron microscopy [2]; (2) X-ray diffraction analysis [3]; and (3) interpretation of mechanical tests in which temperature, stress rate, etc., are varied [1]. In the present paper, the application to the problem of a relatively new technique, that of selected-area electron channelling, is described. The results, although appearing to support the concept of crack-tip plasticity during "brittle" fracture of alumina, are not conclusive in themselves. Consideration of the evidence gathered to date, both for and against plasticity-controlled fracture, suggests experimental approaches that might resolve the issue.

2. Experimental approach

Three-point bend tests were carried out under ambient conditions for two variants of as-fired Lucalox* rod. One set of specimens, broken during a previous fracture mirror–fractographic study, was characterized by a grain size of 15 to 20 μm. The other set was typical of material used in earlier tensile [4] and compressive [5] strength studies in this laboratory, and had a grain size of 24 to 40 μm. The fractured specimens were coated with a very thin layer of carbon, in order to permit conductivity within the SEM. This is a delicate process, since the coating must not be so thick as to destroy, through absorption, the diffraction contrast characteristic of back-scattered electrons.

Electron channelling patterns (ECP) were obtained by operating the SEM in the channelling mode, i.e., basically by rocking the beam about a stationary point within each grain of interest†. For grains in metals [7] and ceramics [6] which are free of plastic deformation, this process generates sharp, fine structured electron channelling patterns; the presence of dislocation damage causes loss of higher order lines, broadening of other lines and, for sufficiently large deformation, the loss of contrast altogether. The spatial sensitivy (beam spot-size) is probably 10 to 15 μm, the effective depth from which channelling information is obtained could extend to as much as 8 μm beneath the surface [8] and, in metals, the technique is sensitive to plastic strains on the order of 0.003 [9].

*From G. E. Lamp Glass Division, Cleveland, Ohio, USA.
†The channelling technique is described in detail elsewhere [6].

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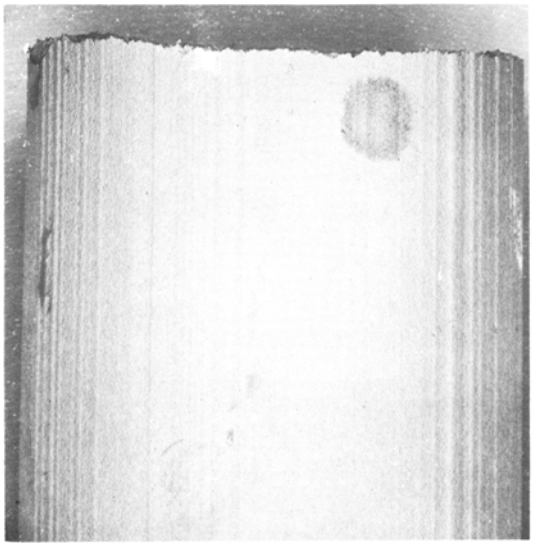


Figure 1 Outer surface of fing-grained Lucalox bend specimen, showing evidence of extrusion (× 11).

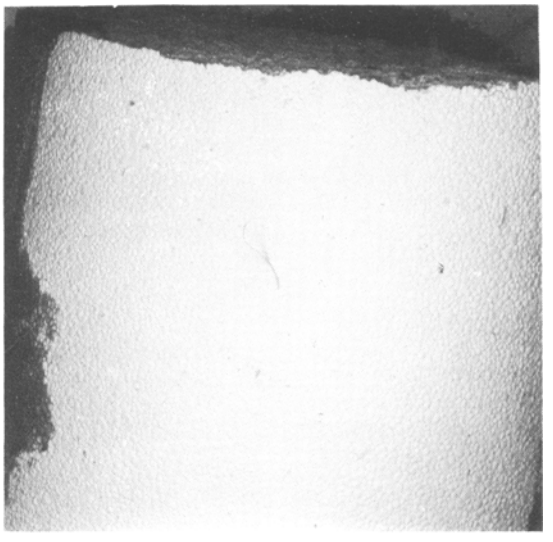


Figure 3 Outer surface of large-grained Lucalox bend specimen; as-fired grains are visible.

3. Results

This section will emphasize the results of electron channelling experiments carried out on the large-grained material since no channelling contrast could be obtained from the fracture surfaces of the fine-grained alumina. Inspection of the outer surfaces of these specimens (see Fig. 1) showed evidence of extrusion. This process appears to have damaged the material to the extent that reference electron channelling patterns obtained from the outer surface were severely distorted.

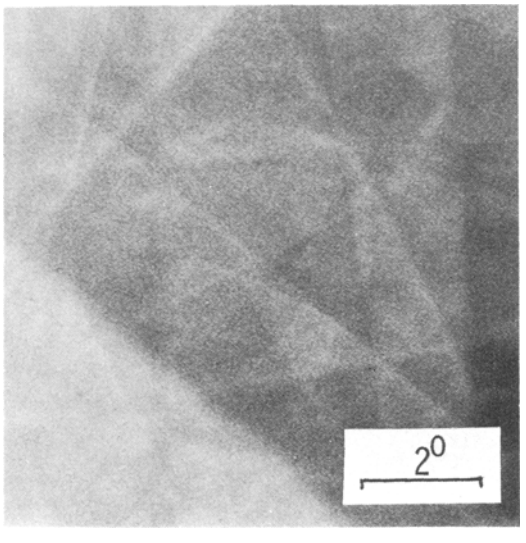


Figure 2 Typical electron channelling pattern from (outer) surface grain of large-grained Lucalox specimen.

On the other hand, Fig. 2 shows a typical channelling pattern obtained from one of the nominally undeformed crystallities comprising the as-fired outer surface (shown in Fig. 3) of a large-grained specimen. All of the numerous outer grains which were investigated yielded patterns of similar quality, i.e., rich in fine structure and higher-order reflections, but slightly hazy due to the presence of the thin carbon deposit. We have obtained similar, but more sharply detailed, results for undeformed SiC [10] which, being a semi-conductor, does not require a conductive surface coating.

An example of a typical Lucalox fracture surface is shown in Fig. 4, where the apparent origin is near Grain 1 (see the arrow in Fig. 4). The numbers within grains in Fig. 4 correspond to the channelling patterns in Fig. 5, while the dots denote grains in which efforts to obtain channelling patterns failed, i.e., the patterns (not shown in Fig. 5) were a uniform grey tone, too diffuse to produce discernible line contrast.

Study of Fig. 5 produces a impression of generally poor electron channelling pattern contrast, with no obvious relationship between pattern clarity and the location of grains relative to the origin. Comparison of Fig. 5 with Fig. 2 indicates that only Grains 5 and 12 (see Fig. 5c and h) yield patterns comparable to the undeformed state. In all other cases, the patterns are broadened, with some (Fig. 5a, d and e) being barely recognizable as channelling patterns.

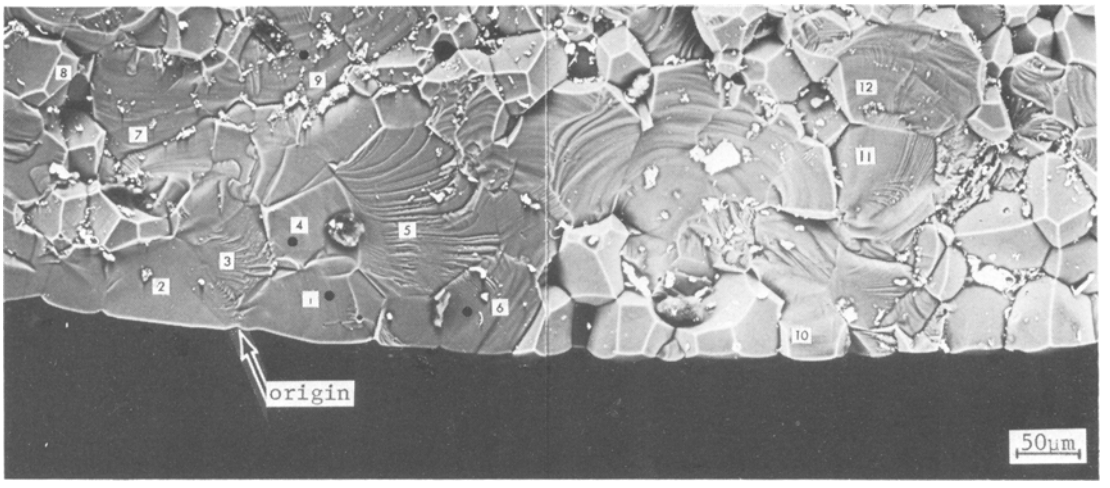


Figure 4 Fracture surface of specimen broken in bending. Dots denote grains in which channelling patterns could not be obtained.

It is interesting to examine the possible relationship between channelling pattern quality and fracture mode. Grains 7 and 12, for example, have almost identical flat, striated fracture surfaces, yet the channelling pattern from Grain 12 is much sharper than that from Grain 7 (see Fig. 5d and h). Near the origin (Fig. 6), Grains 1, 2, and 4 (Fig. 6a and b) are extremely flat and smooth, suggesting cleavage crack growth; nevertheless, the channelling patterns are non-existent for Grains 1 and 4, and very diffuse for Grain 2 (Fig. 5b). On the other hand, Grains 3 and 5 (Fig. 6b and c) are composed of ledges, which might imply [11] enhanced plasticity. Although the channelling patterns produced by Grain 3 (Fig. 5b) are broad and diffuse, those obtained from Grain 5 are very sharp, indicating a minimum of damage. From the foregoing, it seems that fractographic appearance does not offer a clear correlation with the degree of channelling pattern contrast.

4. Discussion

4.1. Interpretation of results

Based on the loss of channelling pattern acuity within most of the grains traversed by a crack, it would appear that plasticity may be associated with crack extension. As of the present, it has not been possible to calibrate plastic strain in ceramics with channelling pattern change, as has been done for metals [9], and the method of defect density assessment is not sufficiently far advanced [8] to assign either a strain value or a defect density to each of the fractured grains. Nevertheless, the line broadening is sufficient to cause one to suspect that a measureble increase in dislocation density should have been affected.

The fractured grains examined in this study exhibit a wide range of distortions and, although most of the channelling pattern lines shown in Fig. 5 are too broad to permit accurate crystallographic orientation determination, they are sufficiently distinct to establish that the fracture planes possess a variety of orientations. Thus, it appears that crystallography may control the degree of channelling line distortion and, hence, by inference, the degree of local plasticity. On the other hand, the lack of correlation between channelling pattern quality and fractographic character suggests that the latter may not be a very reliable method by which to assess the nature of transcrystalline fracture in alumina. It is not obvious, for example, why one should expect lower ECP distortion from Grain 5, Fig. 4, that from the much smoother, more cleavage-like Grain 1.

Several studies by other investigators tend to support the hypothesis that the observed ECP distortion should be interpreted in terms of crack-tip plasticity. Guard and Romo [3], for example, used an X-ray microbeam technique to characterize crystalline deformation below transgranular tensile fracture surfaces of a similar polycrystalline alumina. A highly distorted layer, extending some 10 μm ($\sim$ 1/2 grain) beneath the fracture surface, was detected. Since this zone was present for all crystallographic reflections, it was concluded that

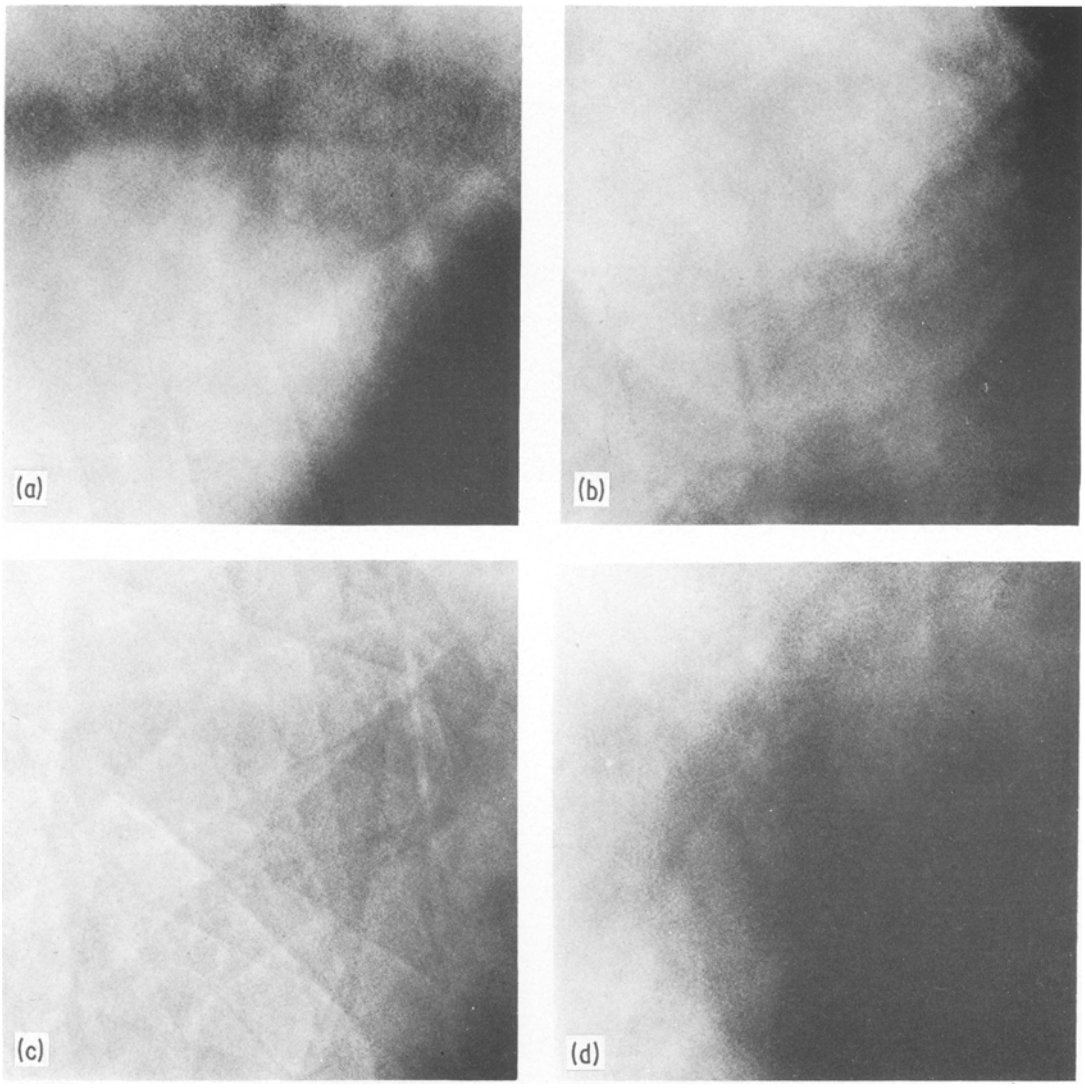


Figure 5 Channelling patterns corresponding to numbered grains in Fig. 4 as follows (a) 2, (b) 3, (c) 5, (d) 7, (e) 8, (f) 10, (g) 11 and (h) 12.

it must have been caused by multiple slip during the passage of the crack.

Congleton *et al.* [1] obtained thin flakes produced during the passage of tensile cracks in Lucalox. These flakes were sufficiently thin to permit the transmission of electrons, and TEM photomicrographs of flakes generated during fracture at 600° C showed evidence of microtwinning and regions of high dislocation density. This temperature is far below that (~ 1200° C) normally associated with dislocation motion in alumina. It should be noted, however, that whether the dislocations observed by Congleton *et al.* [1] were sessile or glissile was not established.

Finally, a study by Pollock and Hurley [12] of strain-rate-dependent fracture strength in sapphire filaments was interpreted in terms of dislocation-assisted crack growth. Evans *et al.* [13] disagreed with this interpretation, and presented arguments for an alternative explanation, based on thermally activated crack growth. In the course of this rebuttal, however, the later authors actually show that the strain-rate dependence of the tensile strength, σ_T, correlates very well with the strain-rate dependence of the dislocation flow stress, σ_f. In particular, it was estimated that, at room temperature, $\Delta \log \dot{\epsilon} / \Delta \log \sigma_f \simeq 24$, while $\log \dot{\epsilon} / \log \sigma_T$ was determined to be 28. (The argument against a dislocation mechanism then reduces to one involving the stress-raising capacity of crack-nucleating

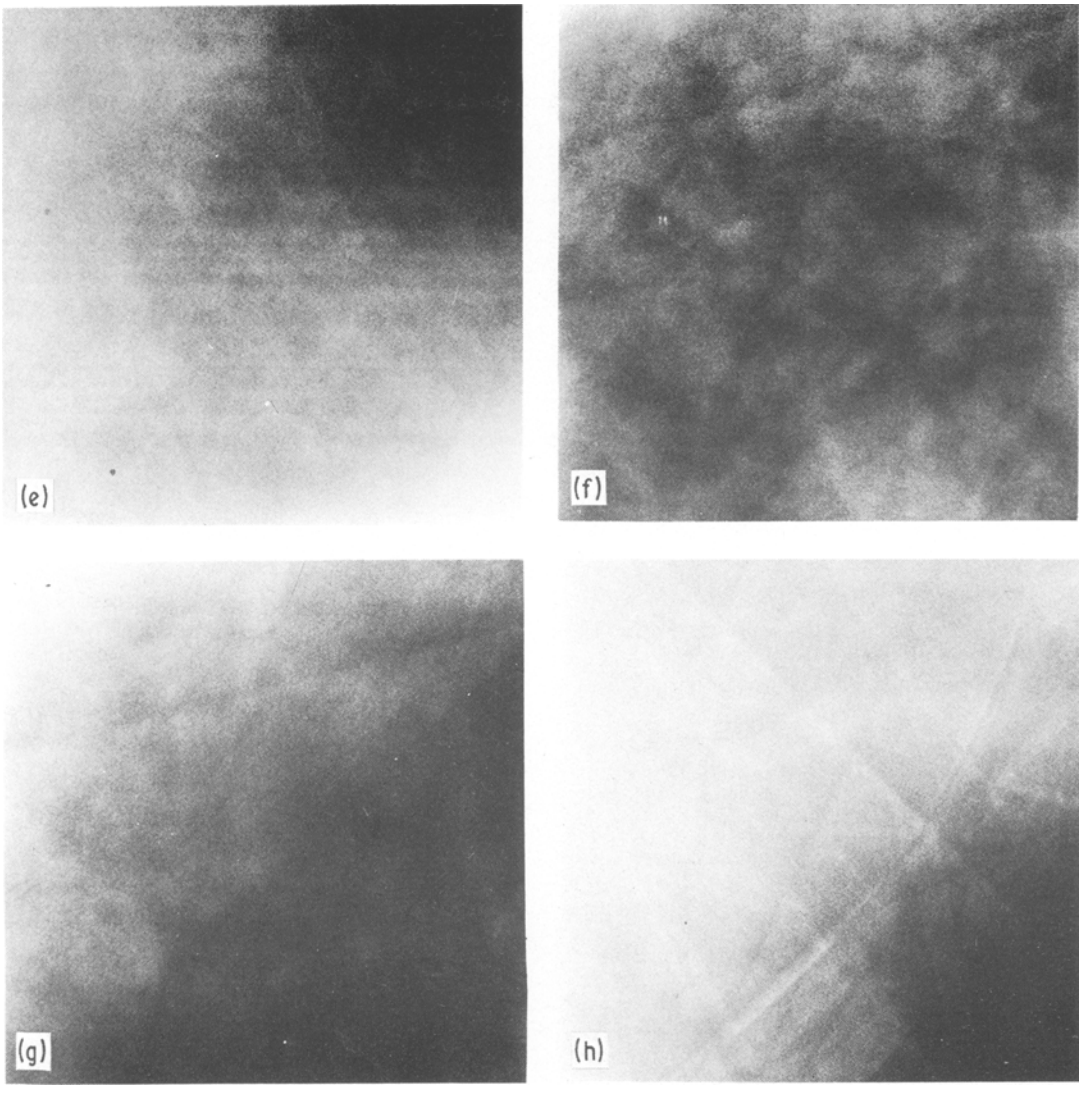


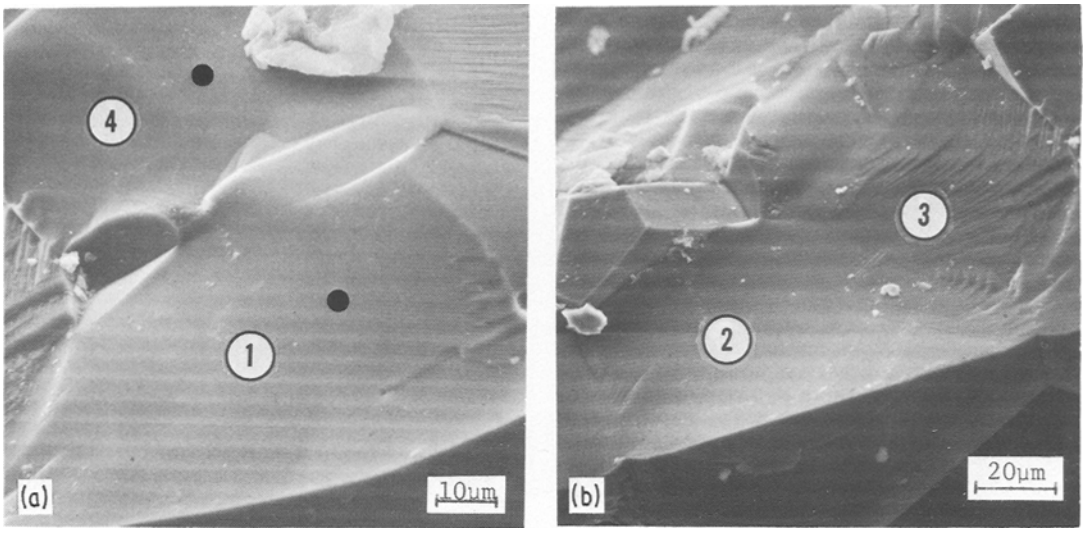
Figure 5 Continued.

voids, and the relative density of nearby pre-crack flaws).

On the other hand, there is at least one very strong piece of experimental evidence which refutes altogether the idea of crack-tip plasticity in alumina. Wiederhorn *et al.* [2] have carried out an exhaustive transmission electron microscope study of arrested cracks at indentations in sapphire and alumina. Cracks of various crystallographic orientations were examined, both at their tips, and back along their flanks. Below 400° C, no evidence of dislocations or microtwins was associated with any portion of the crack trace. It was hypothesized [2] that thermally activated crack growth probably was caused by one of at least three other possibilities: (1) crack growth via stress-induced diffusion of vacancies; (2) fracture via atomic-bond fluctuations at the crack tip; (3) rearrangement of atomic bonds during the fracture process.

The only apparent major difference between various sets of experiments, aside from the techniques employed, seems to be that in the tests leading to the inferred crack-tip plasticity, the cracks studied had been accelerating, while in the indentation crack study by Wiederhorn *et al.* [2] the cracks were arrested. It really is not evident, however, how this difference might account for the observed variance in interpretation.

If it is true that there is no plasticity associated with passage of the crack tip, then the electron



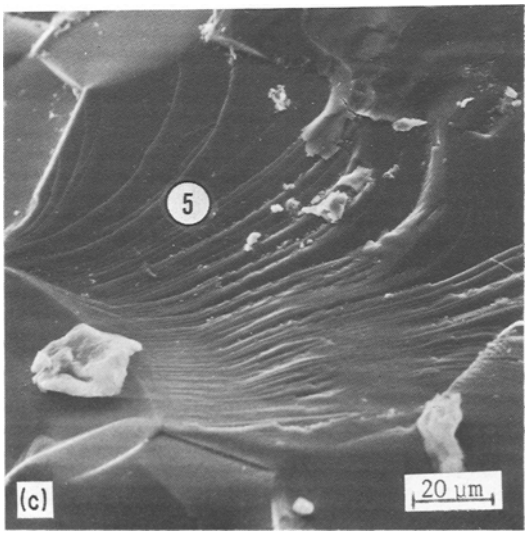


Figure 6 High magnification view of fracture surface near origin. (a) Grains 1 and 4, (b) Grains 2 and 3 and (c) Grain 5.

channelling and X-ray line distortions must be explainable on the basis of some other physical process related to fracture. For example, should the thermal activation process involve vacancy diffusion, then perhaps the observed distortion is due to the presence of local strain fields produced by an excess vacancy population near the fracture surface. Alternatively, the distortion might originate in altered near-surface atomic bonding, induced by passage of the crack. On the other hand, electron channelling should sample [8] crystalline regions lying far below such a hypothetical localized "disordered" region, such that the contribution of the "disordered" region to each channelling pattern is only a small fraction of the total. In this case, fairly high quality diffraction lines would be expected. Similarly, during X-ray experiments of Guard and Romo [3], the "disordered" region would have been removed during sequential etching; yet, it persisted far ($\lesssim 10\,\mu m$) into the specimen.

To resolve this issue, it would be extremely helpful to combine some of the experiments which have been carried out to date. For example, it would be informative to use electron channelling to interrogate the fracture surface of a specimen broken at an indentation pre-crack. Study of the region within the pre-crack, and also outside of it, would correspond to analysis of both decelerating and accelerating cracks in the same specimen. Thin foils of each region for TEM study would provide a direct comparison between TEM and electron channelling. Such experiments are planned.

Acknowledgement

The authors are grateful for the support of the Office of Naval Research under Contract No. N00014-75-C-0668.

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Received 8 October
and accepted 15 October 1981