FRACTURE OF TUNGSTEN SINGLE CRYSTALS AT LOW TEMPERATURES*

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The fracture behavior of both strain-anneal grown and melt-grown tungsten single crystals of high purity was investigated in the temperature range of 77°K and room temperature. It was found that in the absence of surface defects and weak boundaries crystals of all orientations rupture at room temperature by thinning down to a knife edge. At 199°K a transition between rupture and cleavage fracture is observed in some orientations as a result of high strain concentrations at rupture notches. At 199°K and below twinning limits ductility by crack nucleation at the intersection of two twin bands with non-parallel twinning directions. It was observed also that interaction of non-coplanar twins of one family alone could give rise to cracks. The evidence is consistent with the formation of sub-critical size cracks before final fracture which have to await the fulfillment of a fracture propagation condition.

RUPTURE DE MONOCRISTAUX DE TUNGSTENE AUX BASSES TEMPERATURES

Le comportement à la rupture de monocristaux de tungstène de haute pureté, et fabriqués par recuit sous tension et par phase liquide, a été étudié entre 77°K et la température ambiante. Les auteurs ont trouvé qu'en l'absence de défauts de surface, sur les cristaux à contours irréguliers la rupture à température ambiante se fait par amincissement en forme de lame de couteau. A 199°K, on observe une transition entre la rupture et la rupture par clivage, dans quelques orientations dues aux concentrations de déformations élevées aux entailles de la rupture. A 199°K, et en-dessous, le maclage limite la ductilité par nucléation de fissures à l'intersection de deux bandes de macles, avec des directions de maclage non parallèles. Les auteurs observent également que l'interaction de macles non-coplanaires d'une seule famille, peut conduire à des fissurations. La démonstration est en accord avec la formation de fissures de dimensions hypocritiques, qui précède la rupture finale; cette dernière ne peut se produire qu'au moment où les conditions nècessaires pour la propagation des fissures sont remplies.

DER BRUCH VON WOLFRAMEINKRISTALLEN BEI TIEFEN TEMPERATUREN

Das Bruchverhalten durch Reckalterung und durch Schmelzen gezüchteter Wolframeinkristalle hoher Reinheit wurde im Temperaturbereich 77°K bis Raumtemperatur untersucht. Es ergab sich, daß Kristalle aller Orientierungen beim Fehlen von Oberflächendefekten und schwachen Korngrenzen bei Raumtemperatur durch Verdünnung bis herab zu Messerschneidendicke zerreißen. Bei 199°K wird in einigen Orientierungen ein Übergang vom Zerreißen zum Spaltbruch beobachtet, als Folge hoher Dehnungskonzentrationen an Zerreißkerben. Bei 199°K und darunter begrenzt Zwillingsbildung die Duktilität durch Rißbildung am Schnitt zweier Zwillingsbänder mit nicht-parallelen Zwillingsrichtungen. Ferner wurde beobachtet, daß allein schon die Wechselwirkung nichtkomplanarer Zwillinge der gleichen Familie zu Rissen führen kann. Die Beobachtungen sind konsistent mit der Bildung von Rissen subkritischer Größe vor dem endgültigen Bruch, welche die Erfüllung einer Bruchfortpflanzungsbedingung abwarten müssen.

1. INTRODUCTION

The following work on the fracture of tungsten single crystals in the temperature range of 77°K to room temperature is a companion to the preceding study on deformation by the authors; it relates a number of observations on fracture transition and crack nucleation.

2. EXPERIMENTAL PROCEDURE

The method of growth of tungsten single crystal specimens, the details of their preparation, their size, shape, purity and the mode of testing have all been previously described.[1,2] The orientation of the crystals used in this investigation are shown in Fig. 1 of the preceding paper.[2] To prevent premature fracture in the grips it was found essential to carefully electropolish the grip sections of the specimens and have a ratio of grip cross-sectional area to gauge cross-sectional area in excess of two.

Examination of the fracture surface showed that some fractures which occurred prior to yielding started from a corner of the specimen, indicating that a pre-existing surface defect was responsible. The results from these tests were discarded. In all other cases the fracture origins were either internal or occurred on the surface away from the corners.

3. EXPERIMENTAL RESULTS

Observed twinning systems

No twins were observed at room temperature and above. Twins were observed in some specimens at 199°K and in all at 77°K. The twins resulting from the cleavage fracture process were ignored. The most

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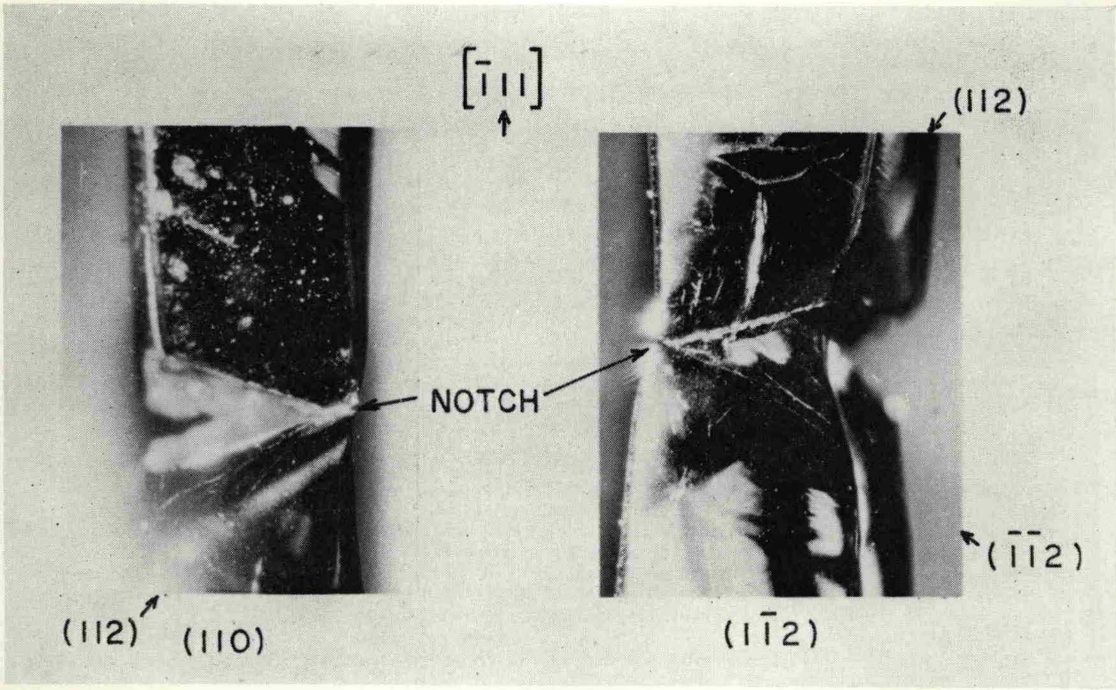

FIG. 1. Rupture to cleavage transition from a rupture notch in a [$\bar{1}$11] crystal tested at 199°K. ×20

favored twinning systems in tension for the corner orientations are listed in Table 1, together with the twinning systems actually observed at 199°K and 77°K; the Schmid and Boas scheme was used to label twins. Some twins were observed on systems on which the applied axial stress produced no shear component and even some twins which should normally be observed only in compression. As these latter twins could have arisen during fragmentation of the specimens they were ignored. The twins with zero Schmid factor, however, could not be dismissed as easily since some of them played a fundamental role in fracture initiation. Specimens which fractured at 77°K containing many twins, were etched to reveal dislocations. Little dislocation multiplication was found, giving the impression that twinning was the dominant mode of deformation at 77°K.

Rupture at room temperature

High flow stresses, accompanied by rapidly decreasing rates of hardening limit the uniform extension in tungsten crystals to about 5–10%. Past this stage a short neck develops and rupture occurs by thinning down to a point or knife edge in all orientations. Crystals in the [011] orientation exhibit the most rapid rate of decrease of strain hardening and therefore the lowest tensile strength, a fact which was first discovered by Goucher.[3,4]

Premature fractures were occasionally observed prior to yielding. The most frequent cause for such fractures were included grains in strain-anneal grown crystals and small unmelted regions in melt-grown crystals. Regions of intergranular fracture with many small hemi-spherical holes were observed on the fracture surface indicating the presence of entrapped

TABLE 1. Best twinning systems and actually observed operative twinning systems in tungsten single crystals in tension

Temperature	Crystal axis orientation	Best twinning systems	Observed twinning systems
199°K	[001]	1, 2, 7, 8	1, 2, 7, 8, 10
	[011]	7, 8, 11, 12	1, 2, 7, 12
	[$\bar{1}$11]	2, 6, 7, 9, 10, 12	2, 7
77°K	[001]	1, 2, 7, 8,	1, 2, 7, 8
	[011]	7, 8, 11, 12	?
	[$\bar{1}$11]	2, 6, 7, 9, 10, 12	7, 9, 10, (1, 3, 5*)

* Compression twins.

gas and the possibility of weak boundaries. The causes of all other premature fractures which started from the outer surface could not be identified.

Fracture at cryogenic temperatures

At 199°K crystals away from the [011] orientation showed considerable ductility and some of them even ruptured. At this temperature crystals around the [$\bar{1}$11] orientation thinned down at the neck and started to rupture at the edges of the necked region. This process produced a sharp notch as is shown in Fig. 1 which eventually caused the rupture to revert to cleavage fracture. Near the [011] orientation the ductility of the crystals decreased considerably, and some fractures occurred soon after yielding. At 199°K twinning was frequently encountered and was responsible for crack nucleation.

At 77°K fracture occurred by cleavage before necking. The fracture stresses for specimens tested at 199°K and 77°K are summarized in Table 2. No significant orientation sensitivity of the fracture strength could be detected. Because of the softness of the machine and the difficulty of observing the entire surface of the deforming specimen it was not possible to detect the formation of twins prior to the final fracture.

Specimens pre-strained at room temperature by 4–5%, when tested at 77°K showed up to 178·5 kg/mm^2 increases in strength above the average value at this temperature.

Crack nucleation at cryogenic temperatures

In a few cases it was possible to identify unambiguously the cause of fracture. Figure 2 shows an example in which two long twins could be traced to the origin of fracture. Alternatively in some fractures although twins could not be found near the fracture, two primary twins could be extrapolated to the origin of fracture, suggesting that partial untwinning may have occurred under the action of released elastic waves during fragmentation. In one [011] crystal strained at 199°K fracture resulted from the intersection of a (112)[$\bar{1}\bar{1}$1] twin with a ($\bar{1}$12)[1$\bar{1}$1] twin as shown in Fig. 3. Since the resolved shear stress on both of these twinning systems due to the applied axial load is zero, they were interpreted to be accommodation twins which have formed to relieve the stresses of the profusion of primary twins in the specimen. A similar case of fracture resulting from accommodation twins was also observed by Edmondson.[5]

Another form of crack nucleation has been observed in [011] oriented crystals in which ($\bar{1}\bar{1}$2)[111] (or equivalent) twins were found to be made up of a row of very short twins staggered with respect to each other in such a way as to subject the narrow ligaments between them to a high tensile stress as shown in Fig. 4(a) and (b). This has then given rise to a profusion of short cracks approximately equal in length to the width of the twin, shown in Fig. 4(c). The small cracks have apparently been blunted before they could extend in a brittle manner. A few such cracks have grown larger extending over the region between two twins eventually resulting in fracture. This type of crack nucleation which does not require a second set of twins with a non-parallel twinning direction has been observed first by Rose[6] in Iceland spar and more recently by Jakovleva and Jakutovic[7] in zinc crystals compressed at 77°K, and by Marcinkowski and Lipsitt[8] in chromium.

Once a crack has been nucleated, propagation of fracture occurred along the (001) planes when these planes were nearly normal to the tensile axis. When the pole of the (001) planes made a large angle with the tensile axis the fracture was somewhat rough in appearance but remained nearly normal to the tensile axis. This suggests that the (001) plane is not a particularly good cleavage plane in tungsten.

When twins were present fracture often propagated preferentially along twin boundaries.

TABLE 2. Summary of fracture data of tungsten single crystals tested at 199°K and 77°K

	Ranges, mean values and standard deviations of		
Temperature	Fracture strength (kg/mm^2)	Fracture strain	Reduction of area
	94.5–308	0–2.36	0–0.906
199°K	$\bar{\sigma} = 161$	$\bar{\varepsilon} = 0.644$	$\overline{RA} = 0.308$
	$\chi_\sigma = 72.3$	$\chi_\varepsilon = 0.9$	$\chi_{RA} = 0.388$
	105.8–209	0–0.292	0–0.253
77°K	$\bar{\sigma} = 159$	$\bar{\varepsilon} = 0.0505$	$\overline{RA} = 0.04763$
	$\chi_\sigma = 26.1$	$\chi_\varepsilon = 0.078$	$\chi_{RA} = 0.670$

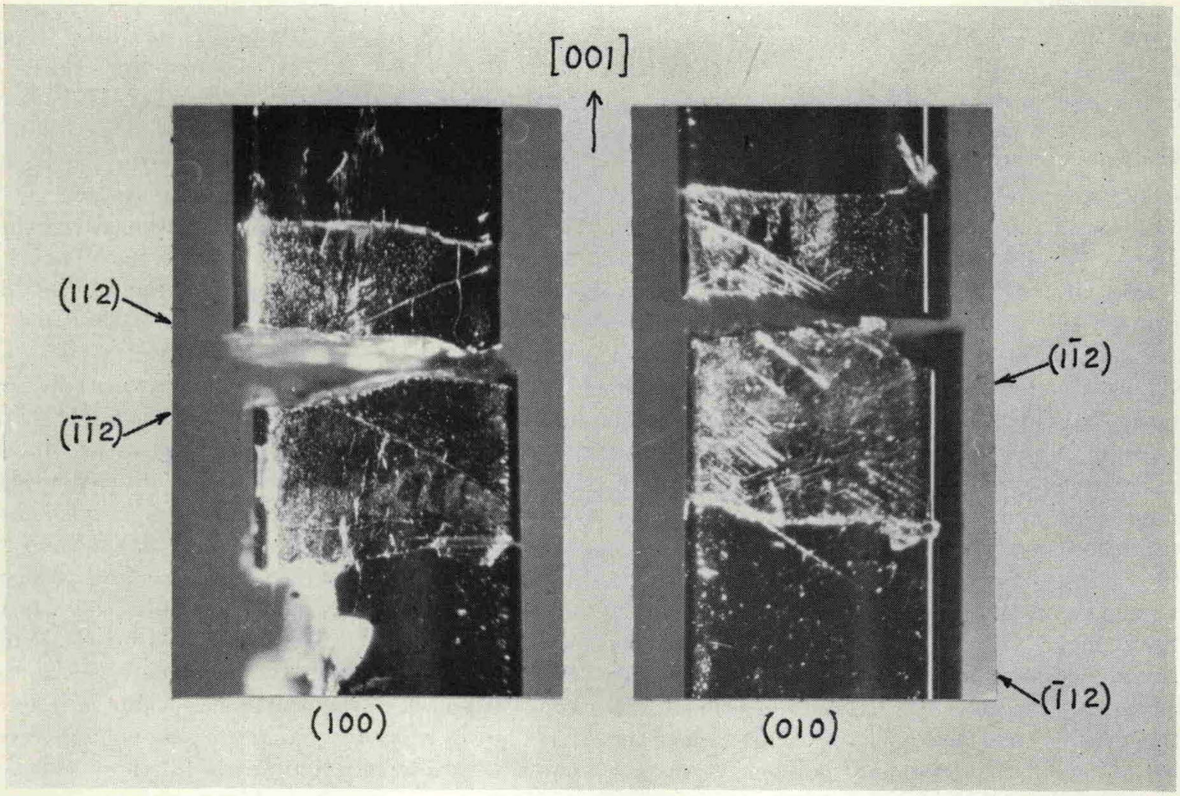

Fig. 2. Crack nucleation from intersection of two non-parallel twins with non-parallel twinning directions in [001] crystal strained at 77°K. × 20

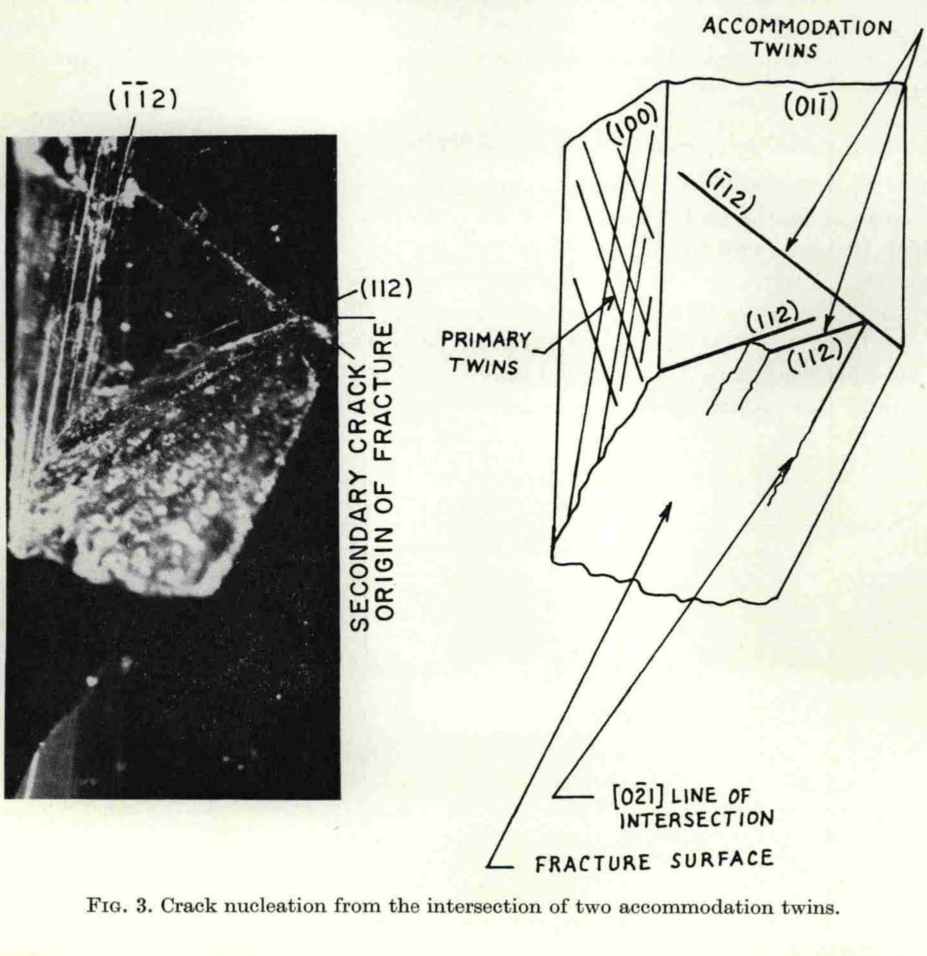

Fig. 3. Crack nucleation from the intersection of two accommodation twins.

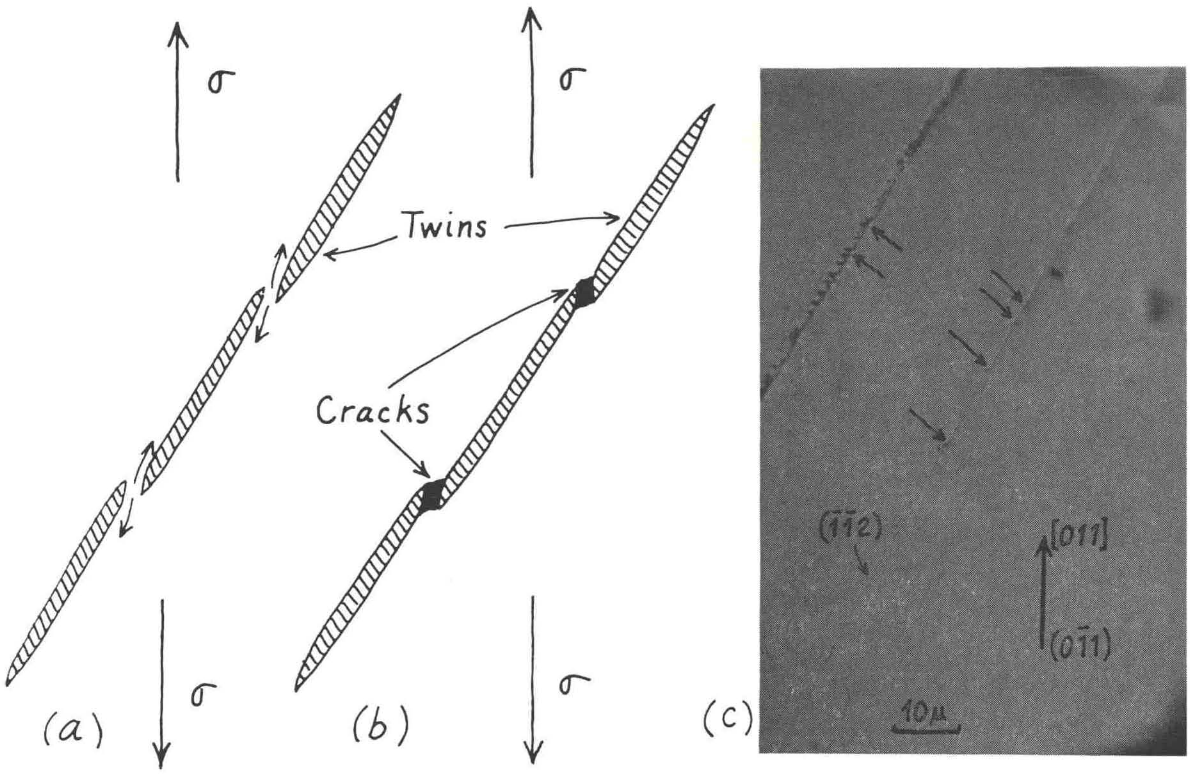

FIG. 4. Crack nucleation from interaction of non-coplanar, parallel twins belonging to the same family in [011] crystal strained at 199°K.

4. DISCUSSION OF RESULTS

The mechanisms of fracture not involving twinning

In the temperature range of this investigation neither slip bands nor any alignment of the etch pits on the surface could be detected in the uniform extension region. Furthermore the dislocation structure in transmission electron microscope specimens was also found to be free of dense dislocation groupings. Thus at room temperature in the absence of twinning it is hard to attribute the occasional premature fractures originating from boundaries of unmelted regions to stresses arising from inhomogeneous deformation. The observed perfect intergranular fractures containing many small hemi-spherical holes rather suggests that these fractures were probably due to poor cohesion across the boundary aggravated by the many small holes. The cause of the poor cohesion could not be identified by electron diffraction from the fracture surface.

Mechanisms of fracture involving twinning

At 77°K the crack nucleation mechanisms unmistakably involve twinning and there is a close similarity in the behavior of tungsten and calcite.[6] Almost all the modes of crack nucleation observed by Rose[6] on calcite involving twins are also found on tungsten.

Rose observed that in Iceland spar cracks often arose from the tips of terminated twins. In tungsten such cracking has not been observed, evidently because the concentration of shear stress at the tip of the twin can be reduced by nucleation of a slip band.

Rose observed bridging cracks between the tips of a series of staggered parallel, non-coplanar twins similar to those shown in Fig. 4. This type of cracking may result from continued re-nucleation of new twins in the high tensile stress region of propagating twins in preference to further extension of already existing twins followed by cracking between the staggered twins. An alternative possibility may be a repeated production of a crack from the tip of a growing twin followed by blunting of the crack by nucleation of a new twin from the tip of the crack. The available evidence cannot distinguish between these two possibilities.

In a third mode of cracking which was also observed by Rose non-parallel twins with non-parallel twinning directions intersect to form a crack. Figures 2 and 3 furnish examples of this best known mode of crack nucleation.

Examination of fractured specimens usually showed a large number of twin intersections which have not produced cracking. This is, no doubt, because cracking has been averted by plastic accommodation such as untwinning of intersections,[9] kinking of the intersection,[10] or slip in the obstacle twin and its surroundings.

Conditions governing final crack propagation

Although there is little doubt that twin-induced crack nucleation is an essential step in the fracture of our tungsten single crystals, it is not at all certain that fracture occurred as soon as cracks nucleated. Evidence of the type shown in Fig. 4 suggests that many cracks which were formed were of sub-critical size and had to wait either for the elevation of stress to propagate them or were extended further by plastic deformation.

If final fracture had been a continuous process with crack nucleation and if twinning can be considered to require a critical resolved shear stress, then resolving the fracture stress on either the most or second most favored twinning systems should give a constant shear stress for all orientations; such a correlation was not found. Furthermore, if final fracture had been simultaneous with the first twinning it would be hard to understand why twinning and fracture occurs after considerable plastic strain in preference to immediate fracture as soon as the lower yield stress is reached in the [011] orientation where the flow stress remains nearly constant. It is rather more likely that twinning occurs early but the nucleated cracks are of sub-critical size and the subsequent plastic strain extends them until they are of critical size.[11] A similar conclusion was reached by Clarke *et al.*[12] for MgO.

If cracks are nucleated by twins they may be expected to be of rather uniform size and of the order of the width of twins. Once such cracks are formed and perhaps extended by further plastic strain, they may propagate when the resolved normal stress reaches a critical value: (a) across the best situated cleavage plane containing the crack or (b) merely when the axial stress reaches a critical value. These two possibilities were explored for our data summarized in Table 2, and good correlation was obtained for both of them. The second possibility, however, is more consistent with the observation that the fracture surface was nearly always normal to the specimen axis.

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