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Fracture and Twinning in Sapphire (α-Al_2O_3 Crystals)

15 MARCH 1963

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UNITED STATES AIR FORCE
Inglewood, California

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(6) FRACTURE AND TWINNING IN SAPPHIRE (α-Al_2O_3 CRYSTALS)

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ABSTRACT

The fracture behavior of 60° oriented sapphire crystals was investigated for both tension and compression. Plastic flow on the basal plane was found to be a factor in reducing the tensile stress required to produce fracture in the temperature range 1100 to 1500°C. Mechanical twins, both (0001) and $(0\bar{1}11)$ types, were produced by compression in the temperature range 1100 to 1300°C. Cracks propagated more readily along the twin boundaries than in the nontwinned crystal.

* degree

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SECTION I

INTRODUCTION

Recent experiments[1-3] have shown that single crystals of alpha alumina (sapphire) can deform plastically, particularly at temperatures greater than 1000°C. The effect of this plastic flow on the fracture behavior of sapphire was not established. However, experiments with other ceramic crystals[4,5] have shown that plastic flow can initiate cracks that lead to catastrophic failure, which suggests by analogy that plastic flow also might be an important factor in the fracture of sapphire crystals.

A recent experimental investigation[3] undertaken to determine the mechanism of plastic flow in sapphire provided an opportunity to study the type of fracture produced after various amounts of plastic shear strain. The results of that study of fracture are reported in the present paper. During the course of this study, it was noted that some of the compression specimens exhibited heterogeneous deformation markings. The crystallography of these markings and their identification as twins are covered also.

SECTION II

EXPERIMENTAL PROCEDURE

Single crystals of sapphire were tested to fracture in tension and in compression at various strain rates and temperatures. Stress and strain at fracture were measured for each test. The fractured specimens were examined optically to determine the mode of fracture propagation.

The specimens were prepared from 0.125-inch diameter, white sapphire grown by the Linde Company by the Verneuil process. The angle between the crystallographic c-axis and the rod axis was within the range of 60 to 70°. The tensile specimens were 10 inches long, with the central two inches having the diameter reduced by grinding to 0.080 ±0.001 inch. The compression specimens were cylinders, 0.125 inch in diameter by 0.5 inch long, produced by sectioning long sapphire crystal rods with a diamond saw. The two flat ends of the cylindrical specimens were ground parallel to within 0.001 inch.

The tensile and compressive forces were applied and measured with an Instron testing machine. The ends of the tensile specimens were held in clamp-type grips to which spring steel straps were attached to provide a universal action of sufficient flexibility to give good axial alignment and to minimize bending. The cylindrical compression specimens were tested by applying a compressive force parallel to the axis of the cylinder by two alumina platens, 0.25 inch in diameter. The surfaces of the specimens and the compression platens were not lubricated. The load was measured to within 0.5 percent by a SR-4 load cell connected to the X-axis of an Instron X-Y recorder. Strain was determined by measuring the crosshead movement with a Baldwin microformer connected to the Y-axis of the recorder, giving an elongation sensitivity of about 10^{-4} inches. The specimens were tested at strain rates from 10^{-6} to 10^{-2} sec^{-1}.

The tensile and compressive tests were conducted within the temperature range of 1100 to 1500°C. These temperatures were produced by a platinum wire resistance tube furnace placed concentrically around the specimen. The temperature was maintained to within ±2°C over the 2-inch gage section of the tensile specimens and over the entire length of the compressive specimens. Estimated accuracy of the temperature measurements varies from ±2°C at 1100°C to ±8°C at 1500°C. All tests were conducted in air.

The fracture surfaces of the specimens were examined optically at 10-60× with a low-power stereomicroscope, and at 50-500× with a research metallograph. A petrographic microscope with crossed polarizers and a back reflection Laue X-ray camera were used to determine crystallographic orientations.

SECTION III

EXPERIMENTAL RESULTS

A. Tension Tests

Each tensile specimen failed by a single crack running across the reduced-diameter portion of the specimen. The plane of this crack was within 15° of the plane normal to the tensile axis. The crack propagation was so rapid that a sharp, explosive sound accompanied each fracture.

The average cross-sectional tensile stress (load/final cross-sectional area) at the time of fracture was not the same for all specimens, ranging from 7 kg/mm^2 to 19 kg/mm^2. The specimens with the largest fracture stresses were those tested at the lowest temperatures (1100°C) or the highest strain rates (> 10^{-3} sec^{-1}), having undergone only a little plastic flow prior to fracture. The specimens with the lowest fracture stresses were those tested at high temperature (> 1400°C) or small strain rate (10^{-6} sec^{-1}), having undergone large plastic strain (up to 60 percent) prior to fracture. The relationship between stress at fracture and strain at fracture is shown in Fig. 1. Table I contains a tabulation of fracture stress, test temperature, strain rate, and total strain to fracture. These data indicate that only strain has any degree of consistent correlation with fracture strength.

Optical examination of the fracture surfaces of the sapphire rods showed that these surfaces contain many ridges and depressions, providing useful information on the origin and propagation of the fracture. A photograph of a typical tensile fracture surface is shown in Fig. 2. The fracture markings are similar to markings produced on the tensile fracture surfaces of glass rods.[6] The smooth area, called the "mirror" area, is at the initial region of the fracture. The curved lines, called "Wallner lines," are a rippling of the fracture surface caused by cyclic alterations of the direction of maximum tensile stress at the tip of the advancing crack; the alterations arise from traveling stress waves originating at secondary crack sources.

The location of the fracture origin, as revealed by the markings on the fracture surface, was related to the crystallographic orientation of the sapphire rods. In almost all specimens, the fracture started where the major axis of the glide ellipse pierced the cylindrical surface of the specimen. Since the orientation of the specimens was such that the slip direction essentially coincided with the projection of the rod axis on the slip plane, the point of fracture initiation occurred at the end of the longest free length of slip path. This geometrical relationship between slip direction and the point of fracture initiation is shown in Fig. 3. Of the 19 tensile fractures examined, only two had points of origin that were definitely not related to the slip direction in the manner shown in Fig. 3. Both of these specimens were tested at high strain rates ($> 10^{-4} sec^{-1}$) at a temperature of 1500°C. Very little plastic shear strain occurred in these two specimens prior to fracture, and a relatively large tensile stress was required to cause fracture.

Propagation of the fractures of the tensile specimens occurred rapidly. There was no evidence of localized necking near the fracture. Neither the general direction of crack propagation nor the small changes in direction associated with the Wallner lines was parallel to any crystallographic planes of low Miller indices. The diameter of the mirror area was not the same for all specimens, but ranged between one-tenth and two-thirds of the diameter of the fractured rod. The spacing between Wallner lines was typically between 10^{-3} and 10^{-2} centimeters. The depth of the depressions between the ridges was about one-half their spacing.

Laue X-ray photographs were taken at the fracture surfaces and at large distances from these surfaces. There was no difference in the amount of spot distortion for the two cases, indicating that there was no localized increase in plastic strain near the fractûre. This suggests that all of the plastic flow occurred prior to fracture, with the rapid fracture propagation occurring in a brittle manner.

B. Compression Tests

The compression specimens failed in several different ways. Specimens tested with small strain rates (10^{-5} sec^{-1}) and high temperatures (> 1400°C) deformed plastically to large shear strains (> 25 percent) without fracturing. One of these highly deformed specimens is shown in Fig. 4. The S-shape of this specimen results from the constraint of slip along the (0001) basal planes by the rigid compression platens. Cracks formed at the two points on the surface having maximum convex curvature, but these cracks did not propagate past the center of the specimen, indicating that the stress was not distributed uniformly in this region. The cross-sectional average compressive stress normal to the end surfaces of this specimen at the conclusion of the test was 13 kg/mm^2.

Specimens tested with high strain rates (> 10^{-3} sec^{-1}) and low temperatures (1100°C) deformed plastically only a small amount prior to failure, by a large number of fractures occurring simultaneously, reducing the specimen to many small splinters. These fractures were approximately parallel to the axis of compression. The average compressive stress at fracture of these specimens was approximately 14 kg/mm^2.

Specimens tested with intermediate strain rates and temperatures deformed plastically and fractured into a few pieces, with the fractures running approximately parallel to the compressive axis. Many of these specimens were particularly interesting, because part of the plastic deformation prior to fracture occurred in distinct bands. In most specimens, these bands were all parallel to the (0001) basal plane. In one specimen, however, the bands were all parallel to the (0001) plane. Both types of bands were identified as twins by both optical and X-ray techniques. These twins were found to be the same as the $(0\bar{1}11)$ and (0001) growth twins that are observed in natural sapphire crystals.[7] Photographs of the twin bands are shown in Fig. 5. A stereographic projection, indicating the position of the twin planes relative to the specimen axis, is shown in Fig. 6.

Optical examination showed that the twinning action produced a displacement of the surface of the crystal. One of the (0001) bands was thick enough (0.3 mm) to measure the displacement direction of the material within the band relative to the material outside of the band. This displacement was distributed uniformly over the thickness of the band and was in a $\langle 1\bar{1}00\rangle$ direction, 30° from the $\langle 2\bar{1}\bar{1}0\rangle$ slip direction of sapphire. The displacement within this band is represented schematically in Fig. 7. The surface of the twinned material had a maximum angular displacement of $9 \pm 1^{\circ}$ relative to the original cylindrical surface. For specimens of this orientation ($\phi = 65^{\circ}$) an angular displacement of 8.5° is predicted by Kronberg's theoretical analysis[8] of twinning in sapphire. Displacement angles could not be measured on the $(0\bar{1}11)$ twins because these bands were too narrow (< 0.05 mm).

The pieces of sapphire containing the twin bands were examined between crossed polarizers of a petrographic microscope. Distinct birefringence color bands were produced by $(0\bar{1}11)$ twins, indicating that the c-axis (a principal optical axis) of a $(0\bar{1}11)$ twin is not parallel to the c-axis of the nontwinned crystal. The (0001) twins did not produce birefringence color bands, indicating that the c-axes of the (0001) twinned and nontwinned crystals are parallel.

X-ray examinations of the twin bands were made with the back-reflection Laue technique. In the $(0\bar{1}11)$ bands, the $\langle 2\bar{1}\bar{1}0\rangle$ directions were parallel in the twinned and nontwinned lattice, while the $\langle 0001\rangle$ directions were displaced by 65°. In the (0001) bands, the $\langle 0001\rangle$ directions were parallel, while the $\langle 2\bar{1}\bar{1}0\rangle$ directions were displaced by 60°.

The twin band interfaces appeared to be weaker than the rest of the crystal, for many (but not all) of the cracks propagated along these planes. A photograph of a fracture fragment is shown in Fig. 8. The right edge of the fragment is part of the original cylindrical surface of the specimen; the other edges are fracture surfaces. Part of the fracture surface follows along the edges of $(0\bar{1}11)$ twin bands, the remainder being conchoidal without following any particular crystallographic direction. This fragment is typical of the fragments produced from specimens containing twins.

SECTION IV

DISCUSSION

The Griffith theory[9] provides a possible explanation of fracture for those specimens that did not have appreciable plastic flow prior to fracture. According to this theory, surface cracks having a depth of approximately 10 microns would be sufficient to cause fracture at the observed fracture stresses near 15 kg/mm^2. Surface cracks of this size may have been produced by the grinding operation used to form the reduced diameter gage length section of the tensile specimens. The scatter in the measured fracture strength of these specimens then could be due either to random variations in the sizes of cracks within specimens or to stress inhomogeneities introduced by small misalignments of the specimens and grips. Flame-polished sapphire rods tested in bending by Wachtman and Maxwell[2] had higher fracture strengths than did the rods of the present investigation, consistent with the assumption that flame-polished surfaces would have smaller cracks than would ground surfaces.

The tensile specimens having large plastic deformation prior to fracture differed from the specimens having only small deformation in two respects: the nominal fracture stress was smaller, and there was less scatter in the value of this stress. The reduction in nominal fracture strength suggests that plastic flow can produce stress concentrations that are more effective in causing catastrophic failure than are the initial small cracks. The fact that fracture was initiated at one end of the longest possible slip length in each crystal that appreciably deformed prior to fracture, suggests that dislocation interactions are important in initiating catastrophic fracture. The reduction in the scatter of fracture strength may indicate either that the effectiveness of the flow-induced stress concentrations is not particularly dependent upon the size of the initial cracks or that the plastic flow can reduce the stress inhomogeneities caused by initial misalignment of the specimen and grips. If reduction of stress inhomogeneities due to initial misalignments were the only result of plastic flow, however, the fracture strength should increase with plastic flow, n ; decrease as actually observed.

Experiments by Brenner,[10] Jackman and Roberts,[11] and Wachtman and Maxwell[2] have shown that the fracture strength of sapphire decreases with increasing temperature above 1000°C. However, only the temperature was varied in these tests; the effect of strain rate was not noted. The experiments of the present investigation showed that the test temperature did not determine uniquely the fracture strength; strain rate also was influential (see Table I). Since both of these factors can affect the amount of dislocation movement that occurs prior to fracture, the results of the present investigation suggest that plastic flow, not temperature, is the important factor affecting fracture strength of sapphire above 1000°C. The corrosive effect known as static fatigue is not expected to be an important factor in aiding crack growth at temperatures greater than 1000°C in low humidity air.[12]

Several theoretical models have been proposed[13] to explain how slip can reduce the fracture strength of crystalline material by specific dislocation interactions. Two of these models are applicable to the fracture of single crystals having unrestrained surface and only one slip plane, such as existed for the present tensile tests of sapphire rods. One model requires the presence of small basal tilt boundaries, the other the presence of small surface cracks.

The possibility of nucleating cracks by the large tensile stress normal to the slip plane at the termination of a tilt boundary within a crystal was first pointed out by Orowan.[14] Quantitative analysis of this mechanism has been considered by Friedel[15] and Stroh.[16] According to their analyses, the tensile stress normal to the slip plane at fracture should be proportional to the reciprocal of the shear stress on the slip plane. Fracture of the sapphire specimens occurred with the tensile stress directly proportional to the shear stress, indicating that tilt boundaries were not the cause of fracture initiation.

Tensile specimens containing small surface cracks will have a large stress concentration at the root of the crack. Griffith[9] has shown that for any particular applied stress, cracks smaller than a critical size will not propagate in an elastic material whereas larger cracks will propagate catastrophically. However, as pointed out by Orowan,[17] subcritically sized cracks may propagate if plastic flow occurs by slip on a crystallographic plane. Orowan's suggested crack mechanism is illustrated in Fig. 9. The stress field associated with a properly oriented edge dislocation lying near the tip of a crack will add an increment of tensile stress to the stress concentration due to the externally applied force. If the dislocation is close enough to the crack tip, the combined tensile stress at the crack tip can be large enough to cause the crack to propagate to the dislocation core. The crack will not propagate beyond the dislocation, however, because the localized stress due to the dislocation will be relieved when the crack reaches the dislocation core. Further crack propagation requires plastic flow to bring another edge dislocation near the crack tip. When this process is repeated a sufficient number of times, the crack can grow in incremental steps until it reaches the critical size required for catastrophic propagation by the Griffith mechanism for the particular value of applied stress.

The fracture behavior of the sapphire tensile specimens is qualitatively consistent with Orowan's fracture model. Slip took place predominantly on closely spaced basal planes making an angle of 20 to 30° with the tensile axis. Surface cracks were probably caused by surface grinding. Fracture took place at lower nominal stress for increasing amounts of plastic strain. Furthermore, fracture after plastic flow invariably occurred at the end of the longest glide path for edge dislocations, which corresponds to the position where the basal dislocations lying parallel to the root of the most highly stressed surface cracks (those having the crack plane normal to the applied tensile force) have a pure edge orientation. In this position, the stress field of a dislocation has the maximum effect on the stress at the root of the crack.

A quantitative analysis of this mechanism can be based upon Griffith's fracture criterion by modifying it to include the growth of a crack by plastic flow. The relationship between fracture stress and plastic strain can be expressed by

$$\sigma_f^2 = \frac{E\gamma/2}{C_o + C(\epsilon)} \quad , \tag{1}$$

where σ_f is the applied stress at fracture, E is Young's modulus, γ is the surface energy, C_o is the initial crack depth, and $C(\epsilon)$ is the increase in crack depth as a function of plastic strain ϵ. The application of this equation requires knowledge of both the initial crack depth C_o and the functional relationship between crack growth and plastic elongation. These two parameters can be estimated by rewriting Eq. (1) as

$$\frac{1}{\sigma_f^2} = \frac{2}{E\gamma}[C_o + C(\epsilon)] \quad , \tag{2}$$

and plotting $1/\sigma_f^2$ as a function of strain ϵ as in Fig. 10. Using values of $E = 30 \times 10^6$ g/mm^2 and $\gamma = 2000$ erg/cm^2, one obtains a value for C_o of 10 microns, with a linear relationship between crack growth and strain up to a strain of about eight percent. Larger amounts of strain apparently did not influence fracture stress. This linear relationship possibly may indicate that crack growth is related to a linear increase in the width of slip bands, and that crack growth stops after the bands completely fill the crystal, at about eight percent strain. A linear growth rate in the width of slip bands for small strain (up to one to two percent) has been observed in LiF,[18] but information on the microscopic distribution of strain within sapphire is not available.

Once a growing crack becomes large enough to spread by the Griffith mechanism without the aid of nearby dislocations, it travels too rapidly to permit dislocation movement to be an effective mechanism for influencing fracture propagation. The crack then advances catastrophically in a completely brittle manner, producing fracture. The fact that the crack did not travel along any one particular crystallographic plane suggests that the direction of crack growth is determined primarily by the stress state at the tip of the crack, not by crystallographic cleavage planes.

The stress and strain distribution produced within the compression specimens was more complex than that in the tension specimens. The lack of lubrication between the specimens and the platens led to large, radically directed frictional forces between the specimens and the platens. The resulting stress state in the specimen thus consisted of radial tensile stresses as well as axial compressive stresses. These tensile stresses were the probable cause of fractures occurring parallel to the rod axis. Specimens that deformed by a large amount to an S-shape (Fig. 4) had cracks at the positions of maximum curvature, suggesting that tensile stress due to bending was present at these places.

The formation of mechanical twins in compression specimens prior to fracture is of particular interest, especially since these twins formed most readily under conditions that permitted only small amounts of plastic flow. Mechanically produced twins, both $(0\bar{1}11)$ and (0001) types, have been reported previously[7,19] as occurring only in sapphires subjected to very large, nearly hydrostatic pressures (greater than 13,000 atmospheres) at temperatures near 25°C. Small amounts of plastic flow also occur under this type of stress condition. This simultaneous occurrence of slip and twinning in sapphire is consistent with Kronberg's theoretical analysis[8] indicating that mechanical twinning in sapphire can be developed from the correct sequence of partial dislocations in successive layers of oxygen atoms.

The fact that twinning occurred in compression but not in tension is expected from the anisotrophy of sapphire crystals. The distance between basal planes relative to the atomic spacing within the planes is slightly less than it would be if the oxygen atoms were in perfect spherical close packing. For crystals with the basal plane oriented 20 to 30° to the specimen axis, twinning will produce a decrease in the total length of the specimen and thus is energetically favorable in compression but not in tension.

SECTION V

CONCLUSION

This investigation shows that plastic flow on the basal plane of sapphire crystals can be a factor in reducing the tensile stress required to produce fracture in the temperature range of 1100 to 1500°C. The observed behavior is qualitatively consistent with Orowan's suggestion that small cracks can grow by interacting with edge dislocations. The amount of plastic flow that may occur prior to fracture depends upon both the temperature and the strain rate; hence, the fracture stress is dependent on these test variables.

Crack propagation in 60° oriented sapphire is brittle and rapid once the Griffith critical size has been exceeded. The crack does not necessarily follow any particular crystallographic plane; usually it produces a surface with patterns of ridges and depressions that are similar to the surfaces produced by the brittle fracture of glass.

Twins may be produced mechanically in sapphire specimens whose rod axis is 60° from the crystallographic c-axis by unilateral compression along the rod axis in the temperature range 1100° and 1300°C at strain rates of 10^{-4} sec^{-1}. Both (0001) and $(0\bar{1}11)$ type twins can be produced. Cracks appear to propagate readily along twin boundaries, suggesting that twin boundaries have a fracture strength lower than the strength of nontwinned crystals.

SECTION VI

TABLES AND FIGURES

Table I. Summary of tensile fracture test data.

Fracture stress kg/mm^2	Temperature ^{o}C	Strain rate sec^{-1}	Total strain
19.1	1495	4.7×10^{-3}	9×10^{-4}
17.1	1242	4.4×10^{-5}	$<1 \times 10^{-4}$
12.8	1379	4.8×10^{-4}	12×10^{-4}
12.4	1480	4.8×10^{-2}	3×10^{-3}
12.1	1089	4.8×10^{-6}	1×10^{-4}
10.5	1204	4.6×10^{-6}	4×10^{-3}
9.5	1240	1.1×10^{-5}	5×10^{-2}
9.2	1364	1×10^{-5}	42×10^{-2}
8.7	1299	3.7×10^{-5}	5.7×10^{-2}
8.3	1394	5×10^{-6}	5.7×10^{-2}
7.9	1294	4.5×10^{-6}	4.3×10^{-2}
7.9	1496	4.5×10^{-4}	7.3×10^{-2}
7.6	1240	1.2×10^{-5}	6.5×10^{-2}
7.6	1197	3.5×10^{-6}	3.4×10^{-2}
7.4	1486	4×10^{-6}	63×10^{-2}
7.3	1392	1×10^{-6}	21×10^{-2}
7.3	1496	4.5×10^{-4}	7×10^{-2}
6.8	1387	2×10^{-5}	11×10^{-2}

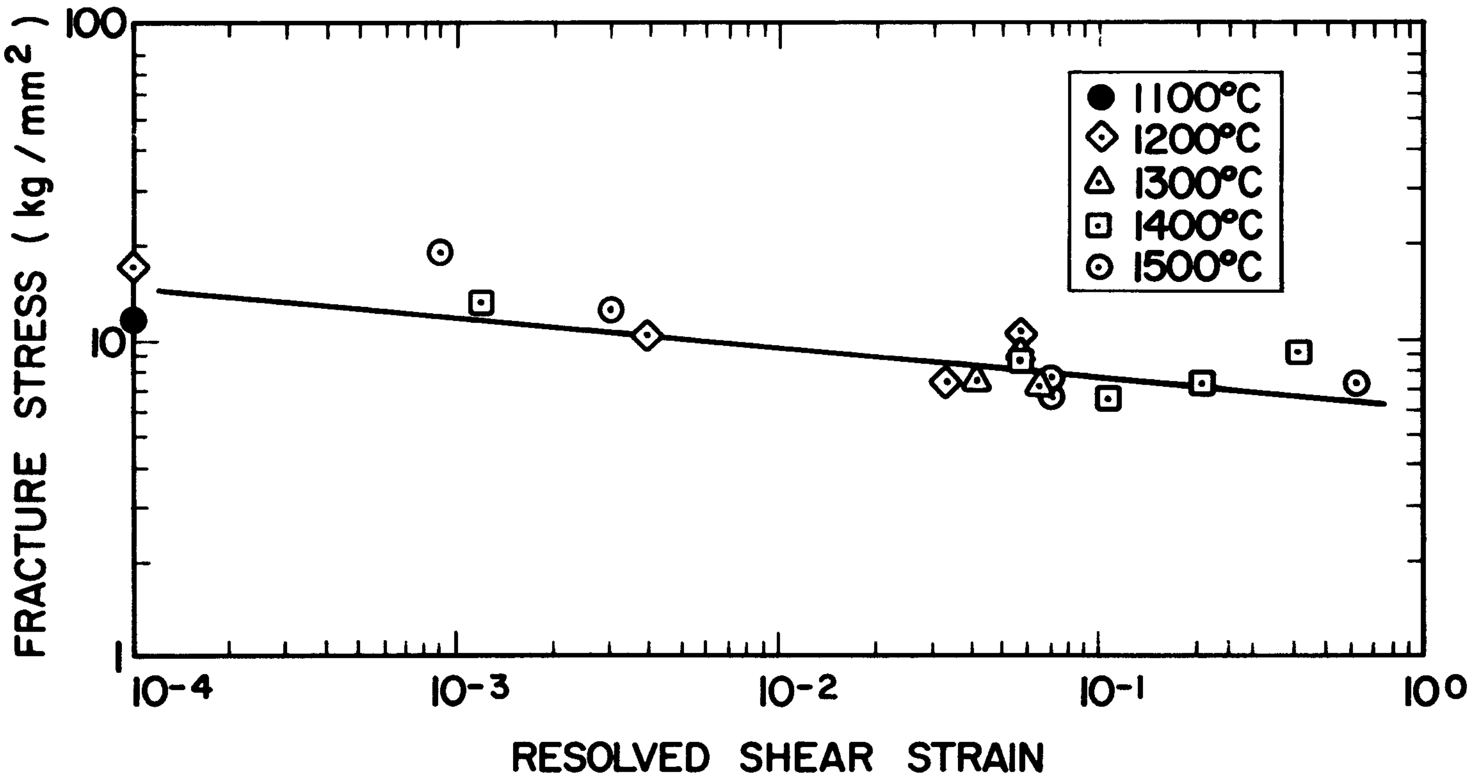


Fig. 1. Variation of fracture stress in tension with strain at fracture.

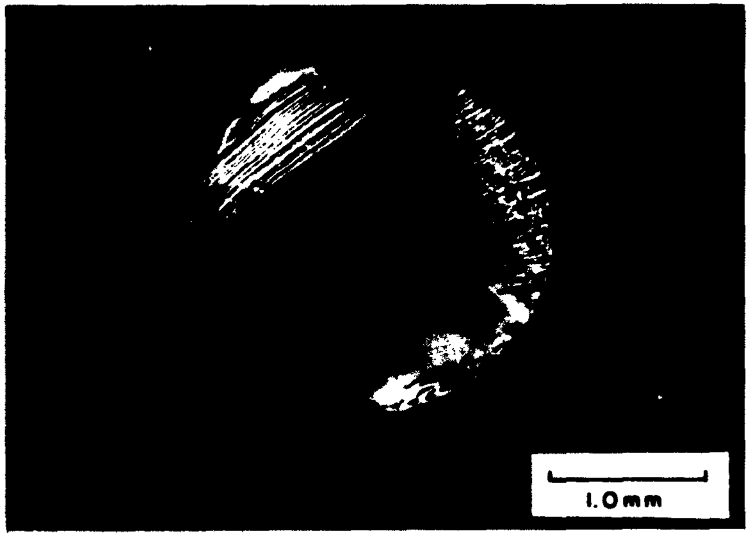


Fig. 2a. Typical fracture surface produced during tensile test.

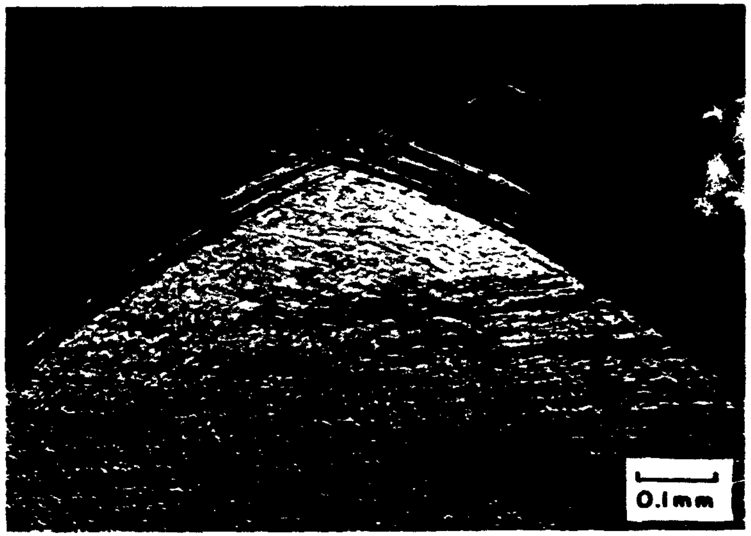


Fig. 2b. Enlarged view of transition from mirror area to Wallner lines on fracture surface at left.

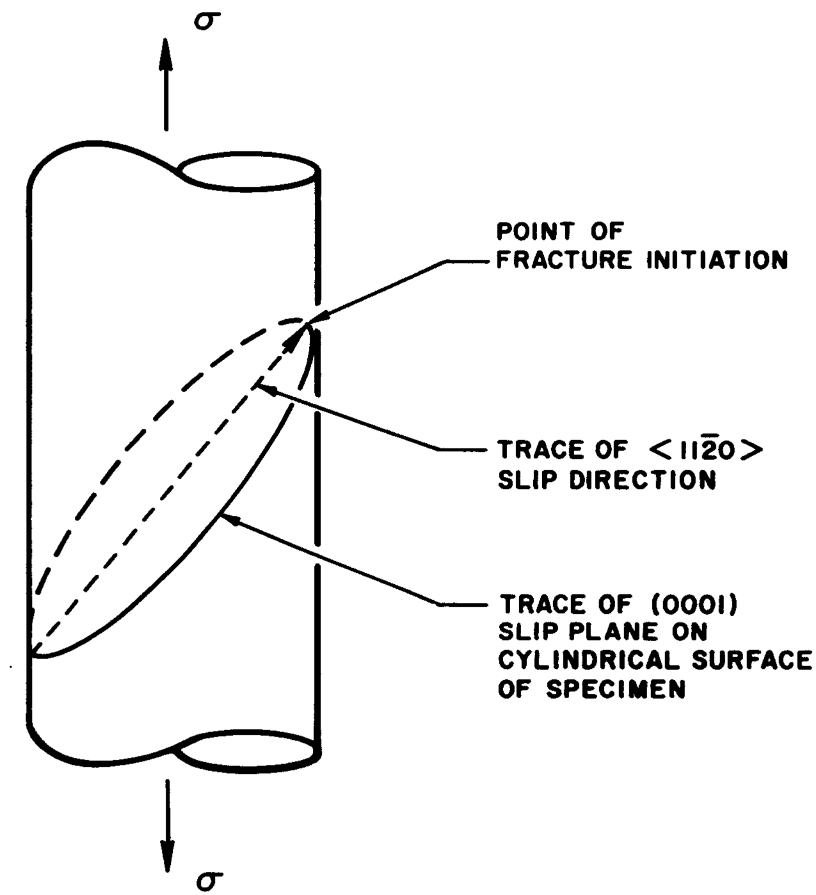


Fig. 3. Relationship between slip direction and point of fracture initiation for tensile tests.

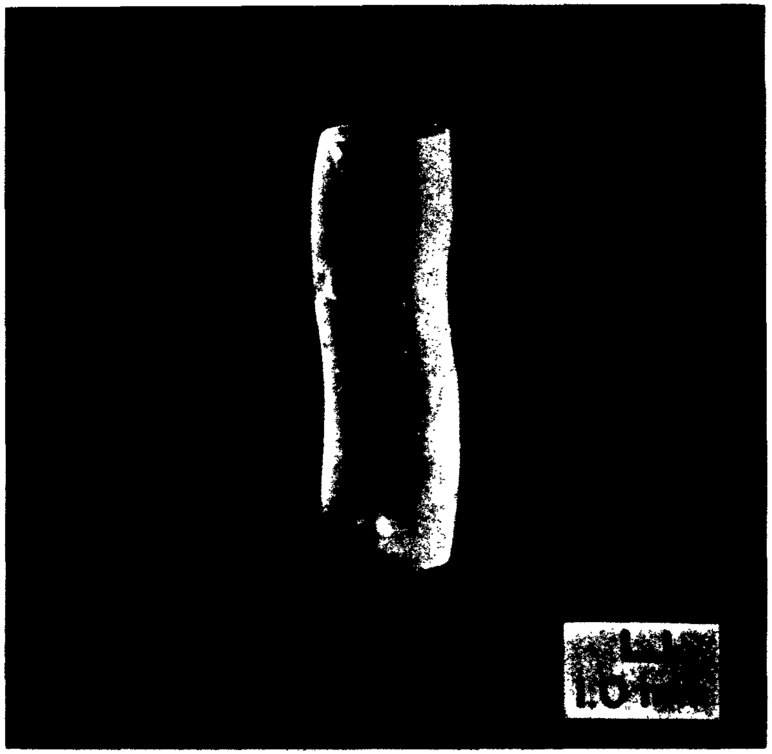
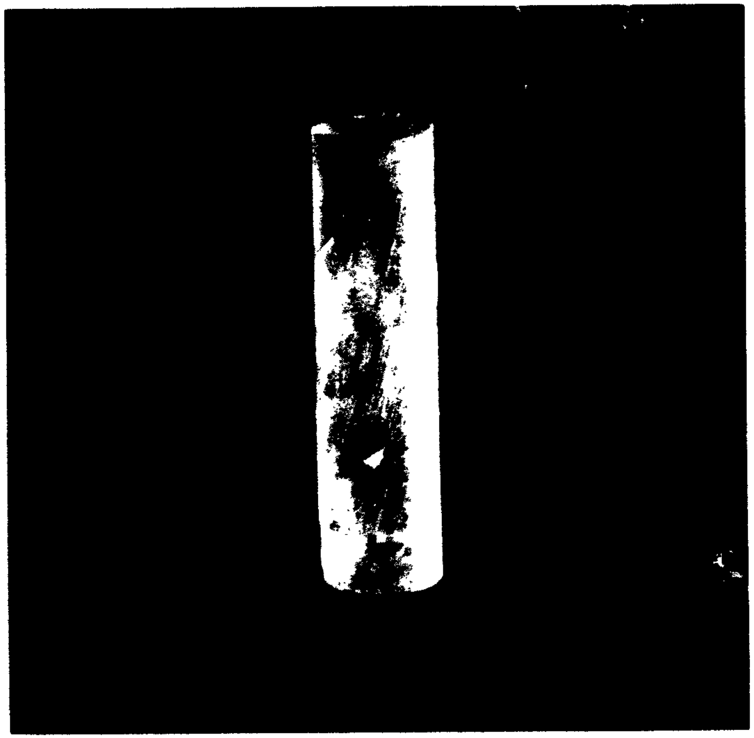


Fig. 4. Plastically deformed sapphire compression specimen.

(0$\bar{1}$11) twins



(0001) twins

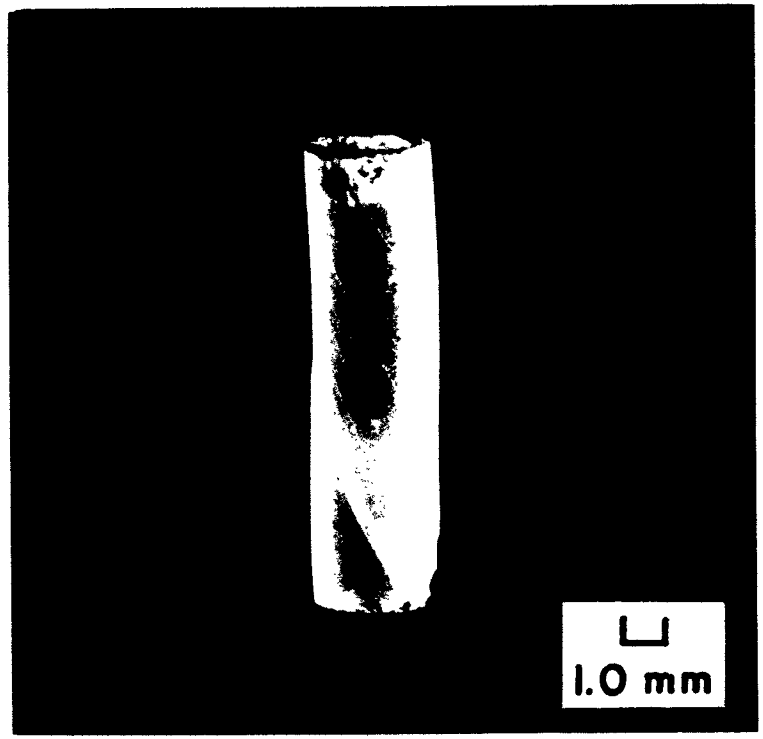


Fig. 5. Twin bands in sapphire compression specimens.

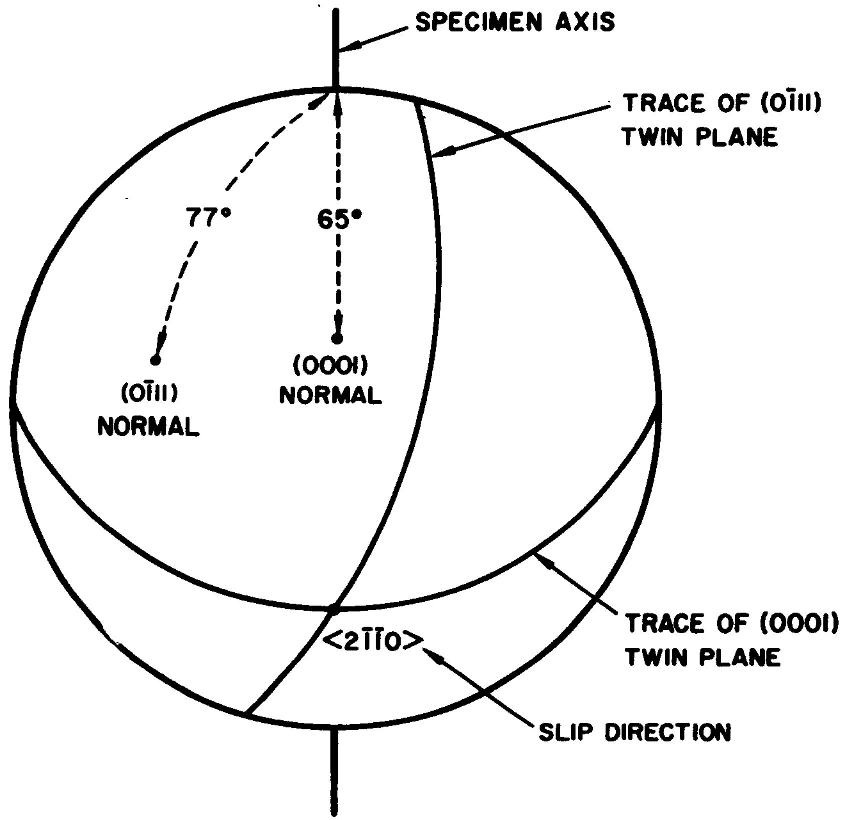


Fig. 6. Stereographic projection showing positions of (0001) twin plane, $(0\bar{1}11)$ twin plane, and $<2\bar{1}\bar{1}0>$ slip direction relative to specimen axis.

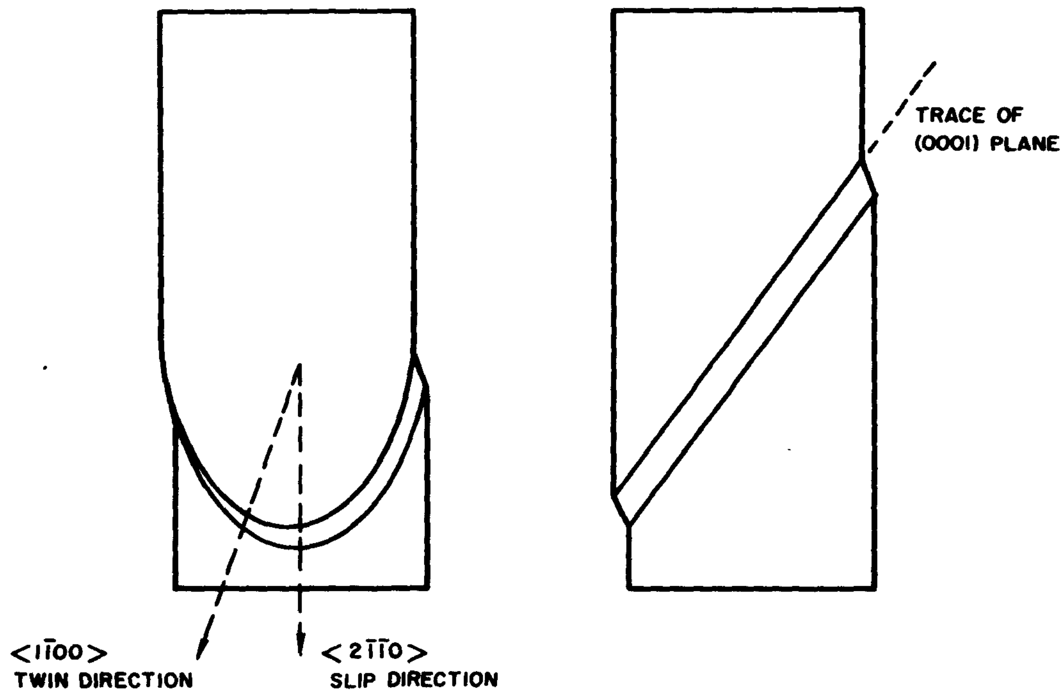


Fig. 7. The (0001) twin band, showing displacement in <1$\bar{1}$00> twin direction.

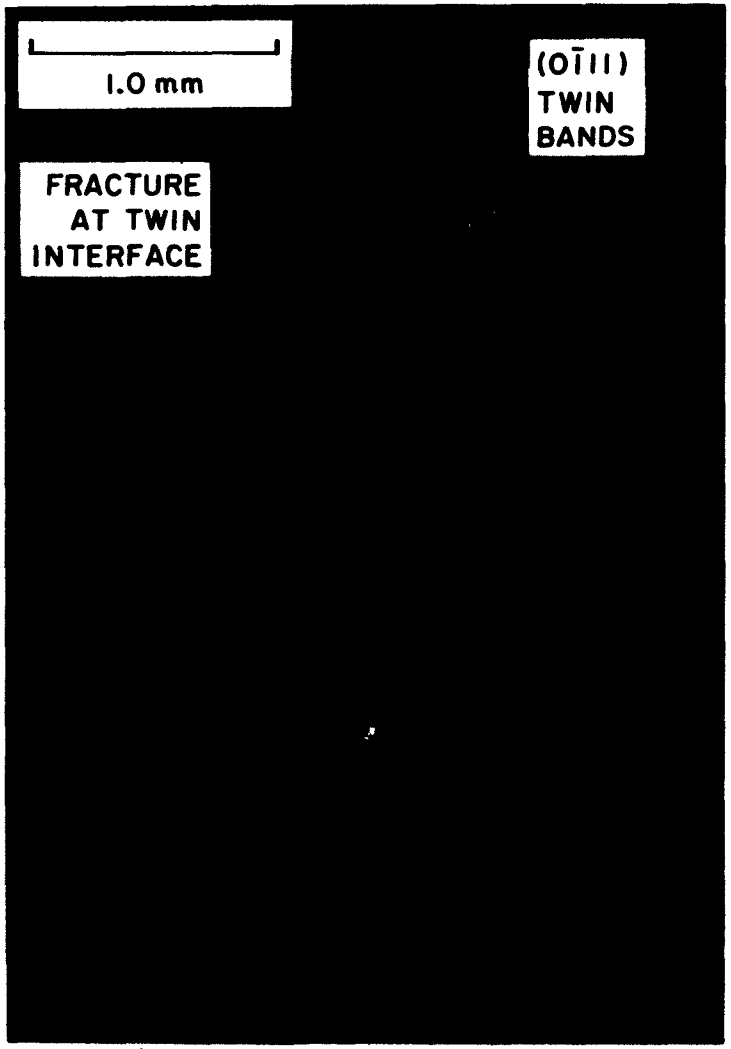


Fig. 8. Fracture fragment of compression specimen containing (0$\bar{1}$11) twins.

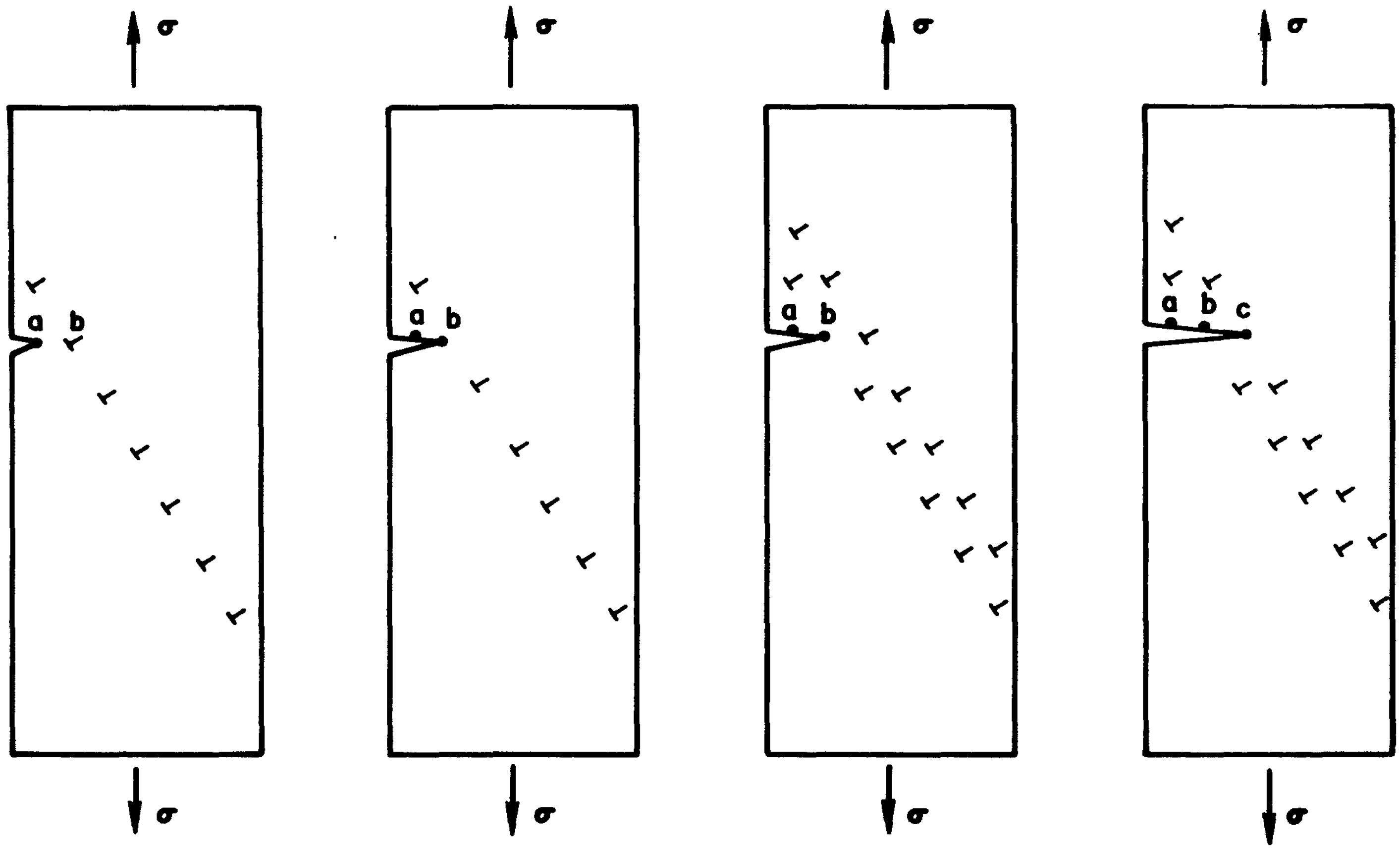


Fig. 9. Orowan's model of crack growth induced by plastic flow.

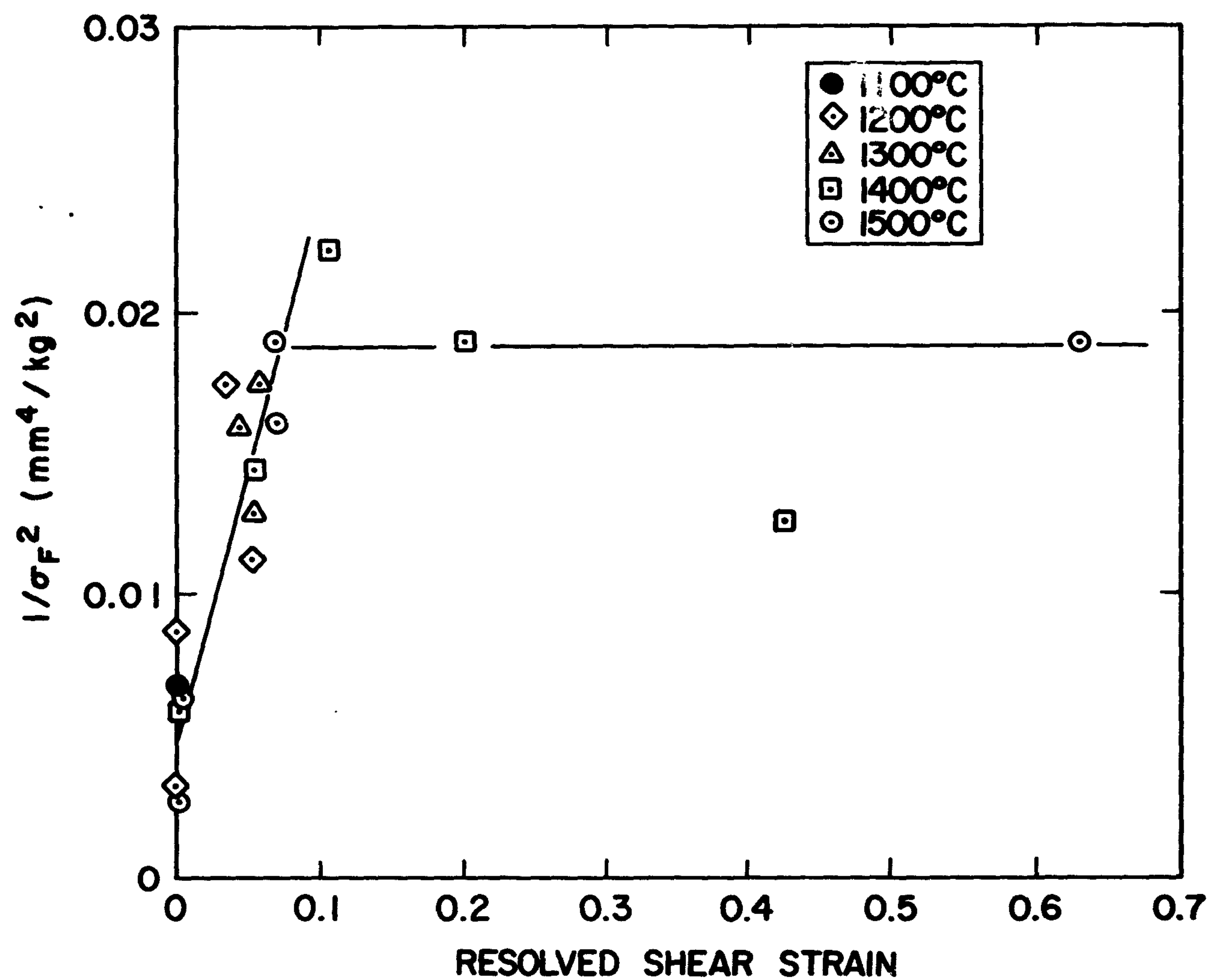


Fig. 10. Reciprocal of fracture tensile stress squared as a function of shear strain

APPENDIX

In addition to the specimens that were tested mechanically in tension and compression, several rods which the manufacturer (Linde Company) previously had broken in bending at room temperature, subsequently became available for examination. Quantitative information on the stress state at fracture was not available. Some of the rods were oriented with the c-axis 60° to the rod axis, others with the c-axis parallel to the rod axis. The fracture surfaces of the 60° rods were similar to those of the 60° tensile tested specimens (Fig. 2); that is, they were primarily conchoidal surfaces modified by Wallner lines and hackle marks, with crystallographic cleavage planes contributing in only a very minor way to the macroscopic appearance. The fracture surfaces of the zero-degree rods were quite different. The fractures followed $(10\bar{1}1)$ planes over most of the surface, with the remainder being conchoidal. An example is shown in Fig. A-1. Optical and X-ray examination did not reveal any twins near the fracture surfaces. The Laue X-ray spots of the zero-oriented rods were more diffuse than were the spots of the 60° rods, suggesting a greater defect structure in the former. An explanation for the difference in the room temperature fracture behavior in bending of the two crystal orientations cannot be offered at this time.

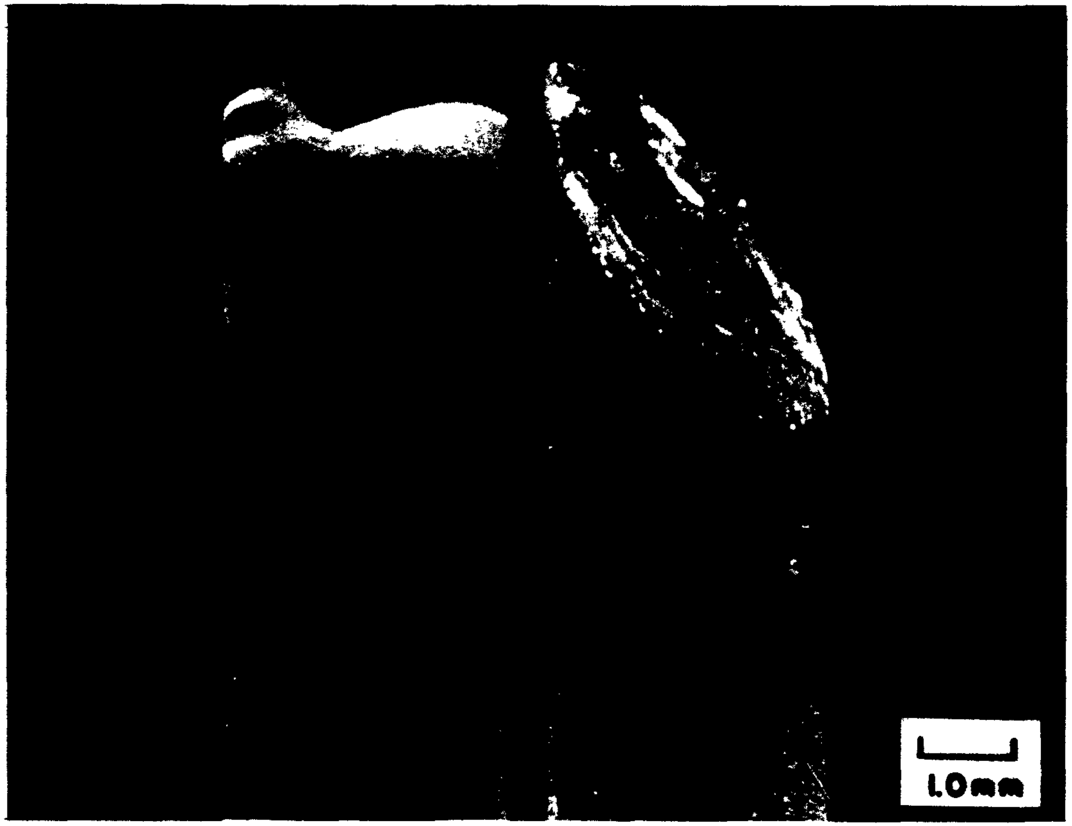


Fig. A-1. Fracture in bending of sapphire rods.

ACKNOWLEDGMENT

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