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Publication Date
1962-08-22
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DEFORMATION AND FRACTURE OF POLYCRYSTALLINE LITHIUM FLUORIDE

William D. Scott and Joseph A. Pask

August 22, 1962
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OF POLYCRYSTALLINE LITHIUM FLUORIDE

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ABSTRACT

Techniques for the fabrication of polycrystalline LiF test specimens were developed and evaluated using single-crystal LiF as a control. An etch was developed which revealed dislocations on all crystallographic faces of LiF. Large grain polycrystalline specimens tested in four-point loading underwent from 0.076 to 0.798% plastic strain before fracture. Deformation was inhomogeneous among the grains because of differences in orientation with respect to the applied stress, and within individual grains because of interactions at grain boundaries. Grain boundaries were barriers to slip, but stresses resulting from slip in one grain were transmitted to neighboring grains and often caused local deformation near the boundary. In one case, local boundary slip occurred on a (010) plane. Three-grain junctions were areas of high residual stresses and fractures originated at boundaries at or near three-grain junctions. Fractures were mixed transgranular and intergranular.
I. INTRODUCTION

The mechanical behavior of ionic materials has been studied both for information on potentially useful structural materials, such as MgO, and for information on the dislocation mechanisms of deformation and fracture which may be common to many materials. A review of the mechanical behavior of ionic crystals has recently been published by Gilman. Of the ionic materials with the rock salt structure which exhibit some ductility, lithium fluoride has been the most extensively studied. Through the use of etch pit techniques, Gilman and Johnston have provided a rather complete picture of the dislocation mechanisms operating in single crystals of LiF. However, because of the marked influence of grain boundaries and neighboring grains on the plastic deformation and fracture of polycrystalline aggregates, one is not able at the present time to predict the mechanical behavior of polycrystals from single-crystal data alone. This study of polycrystalline LiF was undertaken in an attempt to relate the known mechanical properties of single crystal LiF to the deformation and fracture characteristics of polycrystalline LiF.

Feuerstein and Parker have shown that favorably oriented, chemically milled LiF single crystals attain tensile strains as high as 27%. This large strain means that there were no barriers to the emergence of dislocations, and that a slip step was easily formed on the specimen surface. An individual grain in a polycrystalline aggregate, like a single crystal, deforms under an applied
stress, but it must meet the additional requirement that it maintains boundary contact with its neighbors. If a slip band forms in a particular grain, it cannot form a slip step in the surface of that grain unless the neighboring grain deforms elastically or plastically to accommodate the slip step.

Taylor concluded that at least five independent operating slip systems are necessary to maintain boundary contact in a plastically deformed polycrystalline aggregate. In polycrystalline LiF, it is unlikely that five of the six (110) [110] type slip systems normally operative at room temperature are suitably oriented for slip under the applied stress. Investigations on magnesium and on large grained aluminum have shown that boundary contact is often maintained by secondary local deformation near the boundary and that slip in polycrystalline aggregates is inhomogeneous among the grains of the aggregate, and within individual grains.

Because deformation may not be accommodated from one grain to the next, grain boundaries can act as strong barriers to slip. Zener first suggested that cracks could be nucleated by the coalescence of edge dislocations that are blocked in a slip band by some obstacle such as a grain boundary. The theory, as developed quantitatively by Stroh, gives an atomistic description of the relation between plastic flow and crack nucleation. Slip occurs first in the most favorably oriented grains and continues until dislocations pile-up at a grain boundary. High local shear and tension stresses develop at the head of the pile-up, but these stresses are relieved if enough mechanisms of deformation become operative in the original and the adjacent grains. If local deformation does not occur, pile-up will continue until a crack is nucleated.

Barriers to the movement of dislocations can act as fracture sources in LiF. Westwood showed that reaction coatings of MgF₂, or plated metallic coatings, on the surface of LiF single crystals can act as stable barriers to dislocation movement and cause dislocation pile-up, coalescence, and
formation of crack nuclei beneath the film. Feuerstein and Parker observed that grain boundaries in LiF bi-crystals were strong barriers to slip, and that the Zener mechanism was probably responsible for crack nucleation on cleavage planes in bi-crystals. Fractures originating at blocked slip bands have also been observed in single crystals of MgO (which has the same structure and the same slip systems as LiF), and at the boundary in bi-crystals of MgO.

Thus, the evidence from metals and nonmetals indicates that the ductility of a polycrystalline material is controlled by the number of slip systems available to maintain boundary contact and relieve stress concentrations at boundaries prior to fracture. Because of the limited number of slip systems normally operative in LiF, the ductility of polycrystalline LiF should be much less than that of single crystals.

II. EXPERIMENTAL PROCEDURE

A. LiF Purification

A commercial reagent grade LiF was purified by the Bridgman normal freezing method in a graphite crucible in a vacuum furnace at a pressure of 1 μ of Hg. The gradient furnace used has been described elsewhere. After each freezing pass, the colored material containing impurities was broken out of the center of the ingot and the clear material was remelted. Four such passes were required for purification. The uniform single-crystal material thus obtained was used to prepare single-crystal and polycrystal test specimens. Spectrographic analysis of the ingot material and the test specimens indicated 0.002% Mg, less than 0.001% Al, and less than 0.0005% Ca. The amount of Mg impurity in the starting material was also 0.002%.
B. Polycrystal Formation

Polycrystalline ingots were formed by rapidly cooling a rectangular flat-bottomed graphite crucible containing molten LiF. The crucible dimensions were 2 in. long, 2 in. high, and 3/8 in. wide with 1/16-in.-thick walls. Grain size was varied by lowering the crucible through a temperature gradient at rates from 1 to 96 in./hr. LiF has high thermal expansion and low thermal conductivity and is, therefore, sensitive to thermal shock. The minimum average grain diameter obtained with moderate quenches was about 1/8 in. Crystallization started at the bottom and sides of the crucible, and, because of the volume contraction of LiF on freezing, the upper part of the ingots contained large voids. Only the bottom 1/8 in. of each inch-high ingot was used to prepare a polycrystalline test specimen.

C. Specimen Fabrication

Polycrystalline test specimens were cut on a diamond saw using a smooth abrasive blade cooled by water. A polycrystalline ingot was cemented to a bakelite plastic slab with Dekhotinsky cement, and one test specimen 2 in. x 3/8 in. x 1/8 in. thick was cut from the bottom of the ingot. Single-crystal specimens were prepared either by cleaving or by cutting.

Surface damage was removed from cut polycrystals by polishing them 6 to 12 hours in 200 cc of hot (60 to 120°C) concentrated phosphoric acid. The specimen was cooled in the acid and then quickly rinsed in water, ethyl alcohol, and anhydrous ether. About 0.01 to 0.02 in. were removed from each surface. This polish was slightly selective in its attack on different crystallographic faces, but stirring the solution or rotating the specimen on a 1/2-in. radius aided attainment of a macroscopically smooth surface. Single-crystal specimens were polished in a 2% NH₄OH solution, as described by Gilman and Johnston.²a
Specimens were annealed in vacuum in the same furnace used for crystal growth. During annealing, the specimens were held vertically in a graphite rack with only their extreme ends in contact with the graphite. The anneal consisted of heating to $810^\circ C$ for 2-1/2 hours, soaking for two hours and cooling to room temperature in 4-1/2 hours.

D. Bend Tests

Bend tests were carried out in four-point loading on an Instron testing machine. The moment arms were 1/4 in. and the central span was 3/4 in. The crosshead travel was 0.005 in./min in all tests. True stress-strain values cannot be evaluated from bend tests without first having accurate tensile and compressive stress-strain curves for the material under test. In order to compare the mechanical properties of specimens tested in this study, the following approximations were used. A lower limit for outer fiber strain was calculated from the deflection at the load points assuming uniform elastic curvature of the beam between the support points. Likewise, assuming elastic behavior, an upper limit for the outer fiber stress was calculated from the central bending moment and the beam section modulus.

E. Etching and Microscopic Examination

Polycrystalline specimens, after testing, were etched in a solution of $4\% \text{HBF}_4$ in 190-proof ethyl alcohol. The specimen was allowed to rest in the solution without agitation or stirring. The action of the etch depended on the concentration of acid and solution products. Fifty cc of solution were used for each crystal, and etching time varied from 1/2 hour for small single crystals ($1/8 \times 1/8 \times 1$ in.) to 1-1/2 hours for large polycrystals ($3/8 \times 1/8 \times 2$ in.). An evaluation of the reliability of the etch is included under results.
Specimens were examined microscopically by reflected or transmitted light. The light transmission of polycrystals was improved if they were polished for 1 min in Gilman and Johnston’s etch “A”\textsuperscript{2a} either before or after etching in the HBF\textsubscript{4} solution. The use of n-butyl alcohol (index of refraction 1.399) as an immersion medium facilitated transmitted light examination by obliterating the lower specimen surface that was out of focus.

III. RESULTS AND DISCUSSION

A. Etching

The reliability of the 4\% HBF\textsubscript{4} etch used in this study was established by comparing it with Gilman and Johnston’s etch “A” which reveals both fresh and aged dislocations on \{100\} faces of LiF. Figure 1 shows that the same slip bands were revealed by both etches. The same results were obtained when the sequence of etching was reversed. The diagonal lines were edge bands, and the horizontal lines were screw bands. The screw bands were going into the crystal on a plane 45 deg to the plane of the photograph. They were displaced in Fig. 1(b) by an amount equal to the depth of \{100\} surface removed by the chemical polish. The center screw band in Fig. 1(b) appears to split, because the surface was not a perfectly flat \{100\} face. The sub-boundary at the right edge of 1(a) was not etched in 1(b). Some sub-boundaries were etched by the HBF\textsubscript{4} and others were not; the reasons for this selectivity are unknown.

Fig. 2(a) is an enlarged view of the junction at the arrow in Fig. 1(a). The pits show the regular pyramid shape that is characteristic of Gilman and Johnston’s etch “A”. The HBF\textsubscript{4} etch, Fig. 2(b), produced pits that were less regular and less sharply defined, but the general features of the etched bands were the same in both cases.
Figure 3 shows that the same slip bands etched by HBF$_4$ on a (001) face were also etched on a (110) face. The crystal in Fig. 3 was cut on a diamond saw, chemically polished, annealed, and deformed by bending.

A single crystal of LiF was deformed in compression and one end of the crystal was then ground into the shape of a hemisphere, chemically polished, and etched in HBF$_4$. Figure 4 is a picture of the hemispherical end taken looking down the [010] compression axis of the crystal, and showing that the etch is effective on all crystallographic planes.

The orientations of several individual grains in some of the polycrystalline specimens were obtained from Laue x-ray back-reflection pictures. The poles of the planes of these grains that were in the tension or compression surface of the polycrystalline specimens are shown in Fig. 5. These planes were all etched by the HBF$_4$ etch. The figure also shows that there was a random orientation of grains in the polycrystalline aggregates.

B. Single-Crystal LiF

1. Effect of Sample Treatment on Ductility.

Figure 6 is a replot of the Instron load-deflection curve showing the loading characteristics of the four point bend apparatus used in this study. A machine offset of 0.0005 in. occurs at about 0.25 lb. The figure also shows in detail the slight amount of plastic flow that preceded fracture in as-cut single-crystal LiF.

Typical stress-strain curves for single crystals are shown in Fig. 7. The cut samples were prepared by cutting with a diamond saw along four \{100\} cleavage planes. The cutting introduced considerable surface damage, and as-cut specimens were relatively brittle. However, as-cut specimens always underwent a small amount of plastic deformation before fracture. Failure may have been caused either by cracks introduced by sawing, in which case plastic
flow was merely incidental to the failure, or by cracks nucleated by the plastic flow. The second mechanism is believed to be the correct one for the following reasons:

(1) The fracture strength was always high (Av, 2980; range, 2550-3340 psi), whereas a flaw mechanism would be expected to give a few relatively weak samples.

(2) It has been observed by the authors that large cracks were not altered by annealing; but, in every case, the ductility of cut specimens was restored by annealing.

(3) There was an obvious interference to dislocation motion as evidenced by the small amount of plastic flow that occurred at high stresses. The postulated fracture mechanism is that the damaged surface acted as a work-hardened layer that was a barrier to dislocation movement. This surface layer then had an embrittling effect similar to the coatings investigated by Westwood, in which fracture was nucleated by dislocation pile-up under the surface layer.

Removing the damaged surface layer with a chemical polish restored ductility, as shown in Fig. 7.

It has been shown that a low-temperature anneal hardens LiF single crystals. The cut-polished and as-cleaved specimens in Fig. 7 came from slowly cooled single-crystal material. In addition, the cut specimens were heated to about 135 °C for 1 hr while they were being cemented to and removed from the sawing support. The relatively high yield and work hardening of cut-polished specimens, and the similar work hardening of as-cleaved specimens may be attributed to increased hardness of these crystals. A high-temperature anneal reduced the yield and work hardening of these crystals to values similar to those for cut-annealed specimens.

Thus, the present experiments show that different forming methods and heat treatments can introduce large variations in the mechanical behavior of
otherwise similar single crystals of LiF. However, after a suitable high-temperature anneal, all specimens, both cut and cleaved, exhibited similar yield points and ductility. The technique that was then adopted for the preparation of polycrystalline specimens consisted of cutting, followed by chemical polishing and a high-temperature anneal, as described in Sec. II-C.

2. Crystal Orientation and Macroscopic Stress-Strain Curves.

Figure 8 shows stress-strain curves in bending for single crystals of three different orientations. Crystal A was cut with the same orientation as the usual cleaved specimens, B was cut with the side faces (100) and the top and bottom faces (011), and specimen C was cut with its axis close to the [111] direction. A and B were annealed; C was chemically polished but not annealed. The macroscopic stress-strain curves for these crystals indicate large differences. However, as an approximation, Fig. 9 shows that the resolved-stress vs strain curves, where the resolved shear stress is on the appropriate \{110\} \langle110\rangle type slip system, had smaller differences.

The resolved shear stress \(\tau\) was obtained from the calculated outer fiber stress \(\sigma_m\), by the usual equation

\[ \tau = \sigma_m \cos \lambda \cos \theta, \]  

(1)

where the angle between the tension axis (taken here as the specimen axis) and the normal to the slip plane is \(\lambda\), and the angle between the tension axis and the slip direction is \(\theta\). It is noteworthy that a crystal with the cubic NaCl structure has no resolved shear stress on a \{110\} \langle110\rangle type slip system, when stressed in pure tension or compression in the \langle111\rangle direction.

The yield stress was taken to be the point at which the stress-strain curve first deviated from a straight line. The average calculated outer-fiber yield stress for five samples with orientation A was then found to be 1180 psi,
and for four samples with orientation B, 2590 psi. The average calculated
critical resolved shear stresses for these two orientations were 590 and 648 psi,
respectively. Comparing the bulk stress and resolved stress values, it is seen
that the maximum difference in stress between samples A and B is reduced
from 1900 to 200 psi, and between A and C, from 9600 to 950 psi. Resolved
stress values for sample C were obtained because it did not possess a perfect
111 orientation. There was a spread in resolved stress values, due to the
fact that the calculations were approximations because of the complex deforma-
tion pattern realized in bending.

3. Deformation of Polycrystalline LiF

(a) Macroscopic stress-strain curves. Figure 10 shows stress-strain
curves for several polycrystalline specimens. It was found that grain growth
occurred during the anneal. Therefore, to obtain specimens with small grains,
D and E were not annealed. They were cut from two uncemented ingots that had
been cooled on the anneal schedule, chemically polished, and then tested. How-
ever, the grain size was relatively large in all cases and ranged from 11 to 29
grains inside the 1.25-in. support span. There was little variation in grain
size, and no correlation between fracture stress and grain size was observed.

All polycrystalline specimens underwent some plastic deformation
before fracture. Specimens K and L were unusual in the large amount of
plastic deformation preceding fracture. In both specimens, the major part of
the deformation occurred in local areas where one or more favorably oriented
grains extended across the width of the specimen. Therefore it is thought that
the plastic deformation of these two samples was not true polycrystalline be-
havior, but rather owing to local deformation of individual large grains. How-
ever, as will be mentioned below, the influence of grain boundaries on the
fracture of these samples cannot be neglected.
The marked influence of crystal orientation on the shape of the macroscopic stress-strain curve was demonstrated with single crystals. Polycrystalline specimens consisted of many crystals with various orientations. Favorably oriented grains deformed first under the applied stress, but their deformation was hindered by unfavorably oriented neighbors that were not deforming. As pointed out above, a grain oriented in the $\langle 111 \rangle$ direction relative to the applied stress has no resolved shear stress on a $\{110\} \langle 110 \rangle$ type slip system. This fact is significant, because at least one crystal in a polycrystalline aggregate would be expected to have this orientation and thus be unable to deform at all under the applied stress alone. Hence, the resulting macroscopic stress-strain curve of a polycrystalline specimen had little resemblance to a stress-strain curve of a favorably oriented single crystal.

(b) Observations on etched polycrystalline specimens. The etching of deformed polycrystalline specimens revealed that the deformation was inhomogeneous from grain to grain. This behavior is attributed to the various orientations of the individual grains with respect to the applied stress. The deformation was also inhomogeneous within the grains, and local deformation at grain boundaries was often observed.

A photomicrograph of a 3-grain junction in specimen J is shown in Fig. 11(A). The position of the 3-grain junction before the anneal, as delineated by the chemical polish, is in the lower right of the figure. Local deformation may be seen in grain J-2 at the boundary between J-2 and J-3. Figure 11(B) shows the same area under crossed nicols. The light regions are first-order white birefringence, which indicate areas of residual stress. Figure 11(C) is a further magnification of a portion of the horizontal boundary between J-2 and J-3 showing the fine local boundary slip.

Figure 12 shows the orientation of the three grains of Fig. 11(A), and Fig. 13 is a complete stereographic projection for grain J-2. Figures 12 and 13
are plotted with the pole of the tension surface at the center of the projection. The slip traces on the tension surface of the crystal appear on the stereographic projection as points on the basic circle. By measuring the angular positions of the slip traces in the crystal, and comparing them with the positions of the plane traces in the stereographic projection, the slip planes corresponding to the traces in the crystal can be identified. The angles were measured either on a microscope stage or with a protractor on a tracing from a photomicrograph, and the projections were plotted using a 20-cm Wulff Net. Fifty-four slip traces were measured on 14 grains of various orientations and compared with the corresponding plane traces in the appropriate stereographic projections. Fifty measured slip traces were within ±2 deg of the plotted plane traces. Two slip traces were 3 deg in error, and two others were 4 deg in error. A summary of the orientation data for the slip systems is given in Table I, and a numbering system for slip systems of the type [110] <110> is given in Table II.

The fine boundary slip in J-2 shown in Fig. 11(C), corresponds on the stereographic projection for this crystal to the (010) slip plane. The nearest 110 type plane that might correspond to the observed trace is the (101) plane. However, this plane is 15 deg away from the measured position of the slip trace and is outside the range of error in these measurements. A comparison of the (010) and (101) traces for this grain is given in Fig. 11(C).

The two possible slip directions in the (010) plane are [101] and [101]. It cannot be determined from Fig. 13 alone which slip direction was operating. The (010) plane made an angle of 73 deg with the surface of the specimen, and the [101] and [101] slip directions made angles with the slip trace of 65 and 5 deg, respectively. Therefore, at the surface, the dislocations were either primarily screw or primarily edge-type.

Crystal J-3 on the other side of this boundary was very close to the usual {100} cleaved single-crystal orientation. The diagonal slip traces in this crystal
were edge bands while the vertical traces were screw bands. If the displacement caused at the boundary by the edge bands was relieved by local plastic flow in J-2, then the fine boundary slip in J-2 was probably on the system (010)[101], i.e., nearly edge-type. This slip system also had the higher resolved shear stress on it, being, at the fracture stress for this specimen, about 880 psi. For the other slip system, (010)[110], the resolved shear stress was about 540 psi.

Room temperature slip on a {100} plane is difficult in LiF. Gilman has found that for single crystals of LiF tested in torsion, (100)<110> type slip did not occur below about 400 °K. Johnston, on the other hand, has found this type of slip at room temperature for crystals tested in compression; the stress level, however, was about 15 times that necessary for (110)<110> type slip. It is thought that the (010) boundary slip observed in the present case resulted from the combination of two factors: one, a relatively high resolved stress from the applied load, and two, a high concentrated stress at the boundary resulting from large scale deformation of the adjacent grain J-3. Considerable deformation was accommodated between grains J-2 and J-3 without initiation of fracture.

Another feature of this junction was the local slip extending upwards into J-1 from the 3-grain junction, as seen in Fig. 11(A). This slip pattern is similar in appearance to the shear zone predicted by Betteridge and Franklin for grain boundary sliding in creep of metals. That this slip was related to some local stress may be inferred from the very low resolved stress factor (0.13) of the operating systems (J-1, J-5, J-6, in Table I). The slip, like the (010) boundary slip mentioned previously, was confined to a local region of the crystal. Long-range slip on the other operating system (J-1, J-3, J-4, in Table I), which had a high resolved stress factor (0.36), may also be seen in Fig. 11(A).
Figure 14 shows an example of long-range slip from a particular boundary in the compression surface of specimen J. The slip was associated with the diagonal boundary between grains J-4 and J-5, and it was not present along the vertical boundary indicated by the arrow. Table I gives data for the orientation of the slip systems. All the systems with high resolved stress factors were operating in J-5. The systems operating in J-4 had much lower resolved stress factors, all approximately equal. The much denser slip in the system associated with the boundary was probably the result of stresses developed at the boundary by the deformation of the lower grain, J-5. The additional fine slip in grain J-5 at the boundary was probably also due to these same stresses.

Figure 15 shows two heavily deformed grains in the tension surface of specimen L. Both grains were close to the \{100\} type cleaved orientation, and the misorientation between them was a 4 deg tilt. The slip traces running vertically in the grains were screw bands. Gilman found that screw bands were readily accommodated in a boundary in a zinc bicrystal; Fig. 15 indicates a similar situation for a low-angle boundary in LiF.

A more complete picture of the interaction of several grains in an aggregate is given in the data from specimen H in Figs. 16 and 17, and Table I. It was not possible to reconstruct the actual sequence of slip, but several interesting features were observed.

The major slip bands, i.e., the most numerous, heavy, long-range bands present in grains H-1 through H-5 all had a considerable edge component. The slip directions of these systems made angles of from 0 to 28 deg with the slip trace in the tension surface. All but two of the major slip systems, at the fracture stress of this specimen (2280 psi), had resolved shear stresses ranging from 590 to 1100 psi. The lower value is the same as the lower value of critical resolved shear stress obtained for single crystals of this same material. One major system (H-1, H-5) was operating at a resolved stress of 430 psi. The
slip in H-2 which slants steeply down to the right (H-2, H-5) was a major system of almost pure edge type operating under a resolved shear stress from the applied load itself of only 230 psi.

The slip systems operating in H-3 at the boundary between H-3 and H-4, as seen in Fig. 17, were probably accommodating the increased length of this boundary as H-4 deformed. Similarly, the nearly vertical and horizontal slip in H-5 originating at the boundary between H-4 and H-5 is of almost pure edge type and is also accommodating deformation in H-4. The resolved shear stress from the applied load on the latter two systems (H-5, H-1, H-2, in Table I) was 370 psi. Slip of the type present in the curve of the boundary of H-5 was observed in several specimens.

There were several examples of small, local slip systems operating with extremely low resolved shear stresses. The fine horizontal slip in the top and bottom of H-4, and light vertical slip in H-4 near the boundary between H-4 and H-3 (H-4, H-3, H-4, in Table I), had resolved shear stresses from the applied load of only 200 psi. Thus, small local slip systems were made to operate although the resolved stress on them from the applied load was considerably below the yield stress. The additional stress for operation of these systems must have developed from the interaction of deforming grains in the aggregate.

4. Fracture of Polycrystalline LiF

With the exception of specimens I and K, all the fractures of polycrystalline LiF had common features. The fracture went through a 3-grain junction on the tension surface, passed along a boundary for a short distance near the junction, and then moved out of the boundary and into a crystal. The boundary fractures and 3-grain junctions were in the center of the tension surfaces, and the fractures did not originate at boundaries at the outside edges of the specimens.
Typical fractures through 3-grain junctions are shown in Figs. 16 and 18. In the case of specimen H, the fracture probably originated at the boundary near the 3-grain junction. Because of the large deformation of H-4, the region of H-4 near the junction was highly stressed. This condition is indicated by the existence of the horizontal slip operating in this area at a resolved stress from the applied load of only 200 psi. The fracture path branched near the junction; one path followed the boundary and the other path cut transgranularly across the tip of H-4. This behavior also indicates that the part of H-4 near the apex of the junction was highly stressed. Although the macroscopic stress on the specimen at fracture was 2280 psi, the stress on the major slip system in H-2 running into the boundary between H-2 and H-4 was only 590 psi. The fracture may have been nucleated at this boundary by a Stroh mechanism when this slip system yielded.

Nabarro has pointed out that because slip (in metals) occurs only in discrete bands with a spacing of $10^{-4}$ cm, there must be a region about $10^{-4}$ cm thick at a grain boundary where coherence of the grains causes severe elastic distortion and slip on unfavorable planes. Accommodation of the large blocks of long-range slip present in this polycrystalline LiF must also result in elastic distortion and high boundary stresses. Although fractures originated at boundaries and followed a boundary for at least a short distance, this effect is thought to be a result of high boundary stresses rather than an inherent boundary weakness. This conclusion is based on two observations: (a) fractures often moved out of boundaries even though the boundaries were still suitably oriented for fracture under the applied tension, and (b) two polycrystalline specimens containing small amounts of MgF$_2$ (<0.2 and <0.5%) failed with a violent fracture at 13,300 and 10,500 psi respectively, with no apparent plastic flow. The boundaries in these specimens withstood stresses four to five times as great as the fracture stress obtained with pure LiF. Assuming that the MgF$_2$ did not have a large
effect on the strength of the boundaries, then the low-fracture stress of pure LiF cannot be attributed to inherently weak boundaries. It is of interest to note that the fracture strength of the doped specimens was in the same range as the yield stress (≈ 14,000 psi) reported by Nadeau and Washburn for single crystals of LiF doped with about 0.2% MgF₂.¹⁵ A direct comparison is difficult because the hardening effect of this impurity is extremely sensitive to heat treatment. However, even in these strong and apparently brittle polycrystalline specimens, some localized plastic deformation may have been necessary to initiate fracture.

IV. SUMMARY AND CONCLUSIONS

The following points constitute a summary and conclusions from this study:

1. Techniques for forming polycrystalline LiF from the melt and for fabricating polycrystalline test specimens were developed, and these were evaluated using single crystal LiF as a control. The shape of single-crystal stress-strain curves was found to depend on sample preparation and crystal orientation.

2. Large-grain polycrystalline LiF, in the range of 1 to 2 mm, was tested in bending. Some plastic deformation was observed in all cases, but the ductility of polycrystals was much less than that of single crystals. The outer fiber strain (elastic and plastic) ranged from 0.076 to 0.798% at fracture.

3. An etch, consisting of 4% HBF₄ in 190-proof ethyl alcohol, was developed which revealed dislocation slip bands on all crystallographic faces of LiF. The deformation of polycrystalline LiF was investigated using this etch.

4. Plastic deformation was inhomogeneous among the grains of a polycrystalline aggregate. This condition was attributed to differences in orientation of the grains with respect to the applied stress.
5. Deformation was also inhomogeneous within individual grains as a result of interactions between grains. Grain boundaries were strong barriers to slip, in the sense that slip in one grain seldom propagated directly into a neighboring grain.

6. Local deformation along grain boundaries often occurred on \{110\} \langle 110 \rangle slip systems that had resolved shear stresses from the applied load as much as 350 psi below the critical resolved shear stress. In one case, in which conditions were favorable for the development of a highly localized stress condition without the development of a fracture, local boundary slip occurred on a (010) plane.

7. Fractures originated at boundaries close to or at 3-grain junctions and travelled in the boundary before moving into a cleavage plane. Fractures were thus mixed transgranular and intergranular.

8. Grain boundaries were not inherently weak, and they were often able to accommodate large amounts of local plastic flow. It is thought that fractures occurred at grain boundaries where, owing to the limited number of slip systems normally operative in LiF, stresses arising from deformation were not relieved by plastic flow.

At least two possibilities remain for enhancing the ductility of polycrystalline aggregates of materials with ductile properties similar to LiF. One is the suggestion by Parker that extremely small grain size would prevent the development of high local stresses by limiting the length of slip bands within individual grains. A second possibility is that, if the number of potential operating slip systems could be increased from 6 to 12 by somehow promoting easy slip on the system 100 \langle 110 \rangle, then high local stresses might be more easily relieved by local plastic flow. This hypothesis will be tested by deforming polycrystalline LiF at temperatures above 400° K where this second system becomes easily active.
ACKNOWLEDGMENTS

Grateful acknowledgment is made to Professor Richard M. Fulrath for helpful discussions during the course of the research, and to Eugene Kregg for his help in the construction of apparatus.

This work was done under the auspices of the U. S. Atomic Energy Commission.
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FCOTNOTES


Based on a dissertation submitted by William D. Scott in partial fulfillment of the requirements for the degree of Doctor of Philosophy in engineering science, University of California at Berkeley, September 1961.

At the time the work was done the writers were respectively, graduate research engineer and professor of ceramic engineering, Department of Mineral Technology, University of California at Berkeley. W. D. Scott is now research fellow, University of Leeds, Leeds, England.

* Baker and Adamson fluoboric acid, 48-50%, Purified.

† Stress-strain refers to limiting values as described in Sec. II-D.

‡ Personal communication from W. G. Johnston, Research Dept., General Electric Co., Schenectady, N. Y.
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* Table I. Orientation Data for Slip Systems in Individual Grains

**Remarks:**
- Small local slip
- Long slip
- Heavy long slip
- Heavy slip
- Not operating
Table I. (cont.)

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† This system, the alternate possibility to the (010) system, was operating in another part of this grain. The measured angles between the traces of these two systems agreed with the plotted angles of Fig. 13.

* The numbering system for \{110\} \langle110\rangle type slip systems is given in Table II. The \(\lambda\) is the angle between the normal to the slip plane and the tension or compression axis (taken as the specimen axis), and \(\theta\) is the angle between the slip
direction and this axis. "Plane Angle" refers to the angle that the slip plane makes with the tension or compression surface. "Direction angle" refers to the angle that the slip direction makes with the slip trace on the surface. All operating slip systems in a grain are listed in the sequence in which their slip trace appears, in the appropriate figure starting at the vertical and going clockwise.
Table II. Numbering System for Slip Systems in an NaCl Structure.*

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* As they appear in a standard $(001)$ stereographic projection.
FIGURE CAPTIONS

Fig. 1. (a) Slip bands etched by Gilman and Johnston's etch "A" on a (100) face. Reflected light. (b) Same area, chemically polished and etched in 4% HBF₄; film reversed in printing; transmitted light.

Fig. 2. (a) Etch pits from etch "A". Reflected light. (b) Same area, 4% HBF₄ etch. Film reversed in printing. Transmitted light.

Fig. 3. (a) Slip lines on a (001) face and, (b), on the adjoining (110) face, as revealed by 4% HBF₄ etch.

Fig. 4. Slip lines on a rounded end of a single crystal, as revealed by 4% HBF₄ etch.

Fig. 5. Stereographic triangle, showing the orientation of individual grains in polycrystalline specimens.

Fig. 6. Plasticity in as-cut single-crystal LiF.

Fig. 7. Typical stress-strain curves for single-crystal LiF.

Fig. 8. Stress-strain curves in bending, for LiF single crystals of different orientations.

Fig. 9. Resolved-stress vs strain in bending, for LiF single crystals of different orientations.

Fig. 10. Stress-strain curves in bending for polycrystalline LiF.

Fig. 11. (A) Three-grain junction, tension surface, specimen J. The tension direction is labelled T.

Fig. 11. (B) Same area as Fig. 11 (A); crossed nicols.

Fig. 11. (C) A section of the horizontal boundary between grains J-2 and J-3.

Fig. 12. Stereographic projection for the three grains of Fig. 11 (A-B).
Fig. 13. Complete stereographic projection for grain J-2

Fig. 14. Slip from a boundary in the compression surface of specimen J. The compression direction is labelled C. The misorientation in the boundary was 15 deg twist and 30 deg tilt.

Fig. 15. Deformation at small angle boundary in the tension surface of specimen L.

Fig. 16 (A). Slip and fracture in specimen H.

Fig. 16 (B). Identification of grains in specimen H. The misorientations between grains were as follows: H-1 and H-4, 33° twist and 45° tilt; H-2 and H-4, 14° twist and 31° tilt; H-3 and H-4, 29° twist and 22° tilt.

Fig. 17. Detail of slip lines on the tension surface of specimen H.

Fig. 18. Incomplete fracture in the tension surface of specimen F.
Fig. 1. (a) Slip bands etched by Gilman and Johnston's etch "A" on a (100) face. Reflected light. (b) Same area, chemically polished and etched in 4% HBF$_4$; film reversed in printing; transmitted light.
Fig. 2. (a) Etch pits from etch "A". Reflected light. (b) Same area, 4% HBF₄ etch. Film reversed in printing. Transmitted light.
Fig. 3. (a) Slip lines on a (001) face and, (b), on the adjoining (110) face, as revealed by 4% HBF$_4$ etch.
Fig. 4. Slip lines on a rounded end of a single crystal.
Fig. 5. Stereographic triangle, showing the orientation of individual grains in polycrystalline specimens.
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Fig. 7. Typical stress-strain curves for single-crystal LiF.
Fig. 8. Stress-strain curves in bending, for LiF single crystals of different orientations.
Fig. 9. Resolved-stress vs strain in bending, for LiF single crystals of different orientations.
Fig. 10. Stress-strain curves in bending for polycrystalline LiF.
Fig. 11(A). Three-grain junction, tension surface, specimen J. The tension direction is labelled T.
Fig. 11 (B). Same area as Fig. 11 (A); crossed nicols.
Fig. 11 (C) A section of the horizontal boundary between grains J-2 and J-3.
Fig. 12. Stereographic projection for the three grains of Fig. 11(A-C).
Fig. 13. Complete stereographic projection for grain J-2.
Fig. 14. Slip from a boundary in the compression surface of specimen J. The compression direction is labelled C. The misorientation in the boundary was 15 deg twist and 30 deg tilt.
Fig. 15. Deformation at small angle boundary in the tension surface of specimen L.
Fig. 16 (A) Slip and fracture in specimen H.
Fig. 16. (B) Identification of grains in specimen H. The misorientations between grains were as follows: H-1 and H-4, 33° twist and 45° tilt; H-2 and H-4, 14° twist and 31° tilt; H-3 and H-4, 29° twist and 22° tilt.
Fig. 17. Detail of slip lines on the tension surface of specimen H.
Fig. 18. Incomplete fracture in the tension surface of specimen F.
Fig. 18. Incomplete fracture in the tension surface of specimen F.
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