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Berkeley, California

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August 26, 1963

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Recent studies of stress-strain curves obtained in bend tests of large-grained polycrystalline LiF showed that a marked increase in plasticity occurred in the temperature range 350 to 400° C.¹ This effect was attributed to the drastic lowering of the flow stress on the $\{100\}\ \langle 110\rangle$ family of slip systems which has been shown to occur in this temperature range.² It has been found experimentally at room temperature that the $\{110\}\ \langle 110\rangle\$ family of slip systems is unable alone to provide enough independent systems for polycrystalline plasticity,³ and it has recently been shown theoretically that movement on both families is required.⁴

Two sets of experiments have been made in an attempt to study this effect more precisely. Polycrystalline specimens have been studied in compression, which enables more accurate values of stress and strain to be attached to the deformation curves than is the case with bend tests; and compression studies of single crystals have been made in order to determine the flow stress on the $\{110\} \langle 110 \rangle$ and $\{100\} \langle 110 \rangle$ families of slip systems as a function of temperature. Gilman² has published some such curves but did not extend his measurements below 225°C for the $\{100\} \langle 110 \rangle$ family, or above 220°C for the $\{110\} \langle 110 \rangle$ family. Phillips⁵ has published data for the $\{110\} \langle 110 \rangle$ family from -196°C to 600°C but did not study the $\{100\} \langle 110 \rangle$ family.

The polycrystalline specimens were made by crystallization from the melt as previously described^{3,1} and measured approximately 3/16 x 3/8 x 1 in. They were polished in phosphoric acid before testing.^{3,1} The compression apparatus was of the constant loading rate type, and is to be fully described elsewhere.⁶ The load was applied by a calibrated water flow into a bucket attached to a lever system; although constant for a given test, the loading rate varied from about 12 to 20 psi/min. The strain was measured by another lever device: two pivoted, pointed, spring-loaded sapphire rods engaged in two shallow divots 1/2 in. apart in the specimen, and the other ends of the rods operated a linear differential transformer whose output was fed into a pen recorder and used as a measure of strain.

Owing to the large grain size of the polycrystalline specimens it was found that the yield stress depended on the precise orientation of the most favorably oriented grain, and a better quantitative measure of behavior was provided by noting the stress necessary to produce 2% deformation. A plot of this stress as a function of temperature is given in Fig. 1; it can be seen that a distinct step in the curve occurs between 350°C and 400°C and that it tends to extrapolate to zero or a very low stress at the melting temperature of 842°C. The stress-strain curves themselves were similar in appearance to those obtained in bending;¹ the ones at 400°C and higher exhibited a sharp yield, lower work-hardening, and considerably more strain at fracture.

For the single-crystal experiments, two blocks of lithium fluoride^{*} (each $1-1/2 \times 1-1/2 \times 1$ in.) were purchased from the Harshaw Chemical Company, one with {100} faces, the other with the two large faces close

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^{*}A spectrographic analysis of the lithium fluoride specimens indicated impurities of 0.002% Mg, 0.0005% Ca, 0.002% Fe, 0.001% Cu, and less than 0.001% A1.

to {111} orientation. The latter block was reground so that its faces were within 2 min. of arc of the {111} orientation. Specimens approximately 1/4 x 1/4 x 1 in. were cut from each of the blocks with a diamond saw and tested in compression. A specimen compressed in the $\langle 100 \rangle$ direction has a shear stress of one-half of the compressive stress on four of the six {110} $\langle 110 \rangle$ slip systems, whereas a specimen compressed in the $\langle 111 \rangle$ direction (i.e., perpendicular to the {111} faces) has a shear stress of $\sqrt{2}/3$ times the compressive stress on three of the {100} $\langle 110 \rangle$ slip systems. The minimum flow stress which could be measured was about 100 psi because a certain minimum load (corresponding to a compressive stress of about 200 psi) had to be maintained on the specimen to prevent the spring loading of the strain device from pushing the specimen from between the alumina loading rams.

The results obtained are displayed in Fig. 2. For the $\langle 111 \rangle$ oriented specimen ($\langle 100 \rangle \langle 110 \rangle$ slip) the rate of work-hardening was very high, especially at the lower temperatures, and the yields were thus not very well defined. No measurements were therefore made below 200°C for this orientation, but Johnston's observation⁷ that at room temperature the flow stress is about 15 times as high on the $\langle 100 \rangle$ plane as on the $\langle 110 \rangle$ plane is consistent with the present observations. It can be seen that the flow stress on the $\langle 110 \rangle$ plane drops only slowly as the temperature is raised, but that the flow stress on the $\langle 100 \rangle$ plane drops sharply to 400°C and less quickly thereafter. Both curves tend to extrapolate to zero or a very low stress at the melting point, but the flow stress on the $\langle 110 \rangle$ plane.

It thus appears that, on both experimental and theoretical grounds, genuine ductility in polycrystalline LiF cannot be expected until it is

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possible to activate the $\{100\}$ $\langle 110 \rangle$ family of slip systems almost as easily as the $\{110\}$ $\langle 110 \rangle$ family which for this batch of LiF occurred at about 400° C. Also, it appears that the stress vs temperature curves, obtained in compression at a constant loading rate, for both single crystals and polycrystals tend to extrapolate to zero or a very low stress at the melting point.

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FOOTNOTES AND REFERENCES

At the time this work was done the writers were, respectively, lecturer and professor of ceramic engineering, Department of Mineral Technology, University of California at Berkeley. D. W. Budworth is now lecturer, Department of Refractories Technology, University of Sheffield, Sheffield, England.

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FIGURE LEGENDS

- Fig. 1. Compressive stress needed to produce 2% deformation of poly-
- Fig. 2. Flow stress on the $\{100\}$ $\langle 110 \rangle$ and $\{110\}$ $\langle 110 \rangle$ families of slip systems as a function of temperature.



Fig. 1



Fig. 2

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