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Pressure Dependence of Dislocation Mobility in Ionic Crystals[1])[2])

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The mobility of screw dislocations in lithium fluoride single crystals and of edge dislocations in potassium chloride single crystals has been measured in pure bending tests under hydrostatic pressures of 1 bar and 4.3 kilobars, using the repeated etching technique. The increase of pressure has no effect on the velocity-stress relationship: similarly, no pressure dependence is observed in macroscopic deformation tests conducted under comparable conditions on the same materials. The results show that point defect formation does not control the dislocation motion in these crystals; comparison is made with earlier work, and the effect of impurity distribution on the pressure dependence of dislocation mobility in lithium fluoride is discussed.

Die Beweglichkeit von Schraubenversetzungen in Lithiumfluorid-Einkristallen und von Stufenversetzungen in Kaliumchlorid-Einkristallen wurden in reinen Biegungsuntersuchungen unter hydrostatischen Drucken von 1 bar und 4,3 kilobar gemessen, wobei Wiederholungsätztechnik angewendet wurde. Druckanstieg hat keinen Einfluß auf die Geschwindigkeits–Dehnungs-Beziehung; in ähnlicher Weise wurde keine Druckabhängigkeit in makroskopischen Deformationsuntersuchungen, die unter vergleichbaren Bedingungen an denselben Materialien geführt wurden, gefunden. Die Ergebnisse zeigen, daß Punktdefektbildung die Versetzungswanderung in diesen Kristallen nicht steuert. Ein Vergleich mit früheren Arbeiten wird durchgeführt und der Einfluß einer Defektverteilung von der Druckabhängigkeit der Versetzungswanderung in Lithiumfluorid diskutiert.

1. Introduction

Davis and Gordon [1] have shown that the flow stress of lithium fluoride single crystals tested in compression is unaffected by increasing the hydrostatic pressure from 1 bar to 4.3 kbar at strains between 0.25 and 3.0%. They did, however, observe increases of up to 30% in the flow stress of other alkali halides on increasing the hydrostatic pressure to 4.3 kbar: the increases in general were found to be proportional to the change in elastic constants of the alkali halides with pressure.

Haworth, Davis, and Gordon [2] measured dislocation velocities by the etch-pit method and conducted stress–strain tests on lithium fluoride single crystals, finding that neither the stress τ_v required to produce a given dislocation velocity, nor the macroscopic flow stress τ_f, were affected by increasing the hydrostatic pressure.[4]) These results are consistent with the microdynamical theory of crystal plasticity [3] in which it is assumed that τ_f and τ_v are linearly related. However, the macroscopic tests were carried out in compression whereas

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[4]) The applied stresses reported in the paper [2] are in error; corrections are given in the Appendix and Fig. 1 of the present paper.

the dislocation velocity measurements were made on specimens deformed in four-point bending. Furthermore, the functional dependence of velocity on applied stress was not determined under pressure. These experiments have now been extended to cover dislocation velocities over a range of four orders of magnitude at both 1 bar and 4.3 kbar, and to include, for comparison, macroscopic tests in four-point bending at 1 bar, 4.3 and 7.0 kbar. Both the flow stress tests and the velocity measurements were made on crystals of given impurity concentration which were prepared in identical fashion. Similar experiments were conducted on potassium chloride single crystals.

The experimental techniques used have been described in detail elsewhere [1, 2, 4, 5]. The lithium fluoride crystals tested by Haworth et al. [2] contained less than 20 ppm total cationic impurities, and are designated "type A". Impurity analyses of the crystals used in the present work are given in Table 1. Lithium fluoride crystals were γ-irradiated in a Co^{60} source for approximately one hour (0 to 1 Mrad/h) to facilitate cleavage into bending specimens, then tested as irradiated or after annealing for two hours at 450 °C. Potassium chloride crystals were cleaved as-received, then annealed six hours at 700 °C before testing.

Table 1

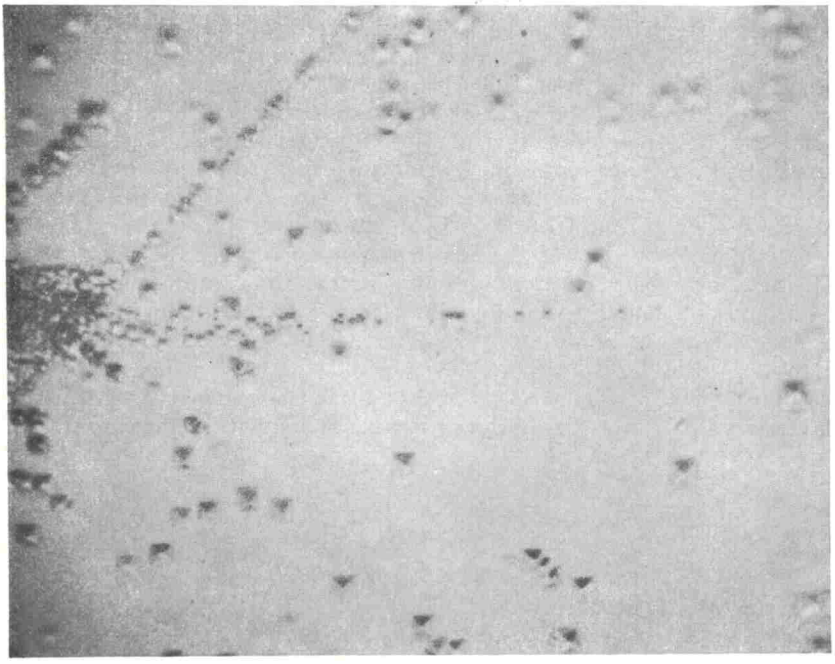
Impurity analysis of crystals tested (Figures are ppm weight, $\pm 20\%$. Spectrographic analysis performed by Bridgeport Testing Laboratory, Bridgeport, Connecticut)

Impurity	LiF (type B)	KCl
Calcium	1.0	1.0
Magnesium	1.2	1.0
Iron	6.0	5.2
Zinc	2.2	0.8
Aluminum	2.8	1.4

2. Results

Inspection of the etch-pit patterns showed that dislocations are not generated or moved under the influence of the applied hydrostatic pressure alone, and that the same mode of deformation occurs at both atmospheric pressure and high pressure on bending the crystal.

The motion of dislocations at 1 bar and at 4.3 kbar in the same lithium fluoride crystal is illustrated in Fig. 1 a. The crystal was etched to reveal dislocations, stressed at atmospheric pressure to move the dislocations, re-etched, then stressed again at 4.3 kbar and etched a third time. The applied stress and loading time were the same at both pressures. The movement of an array of screw dislocations in the [010] direction (i.e., from left to right), from a rosette fault on the (100) face of the crystal, is revealed in the photomicrograph, in which three pit sizes can be distinguished. In Fig. 1 b the same area is shown after restressing at atmospheric pressure, again at approximately the same load and time as before, and etching once more. Further movement of dislocations in the [010] direction is revealed, as well as some additional damage caused by



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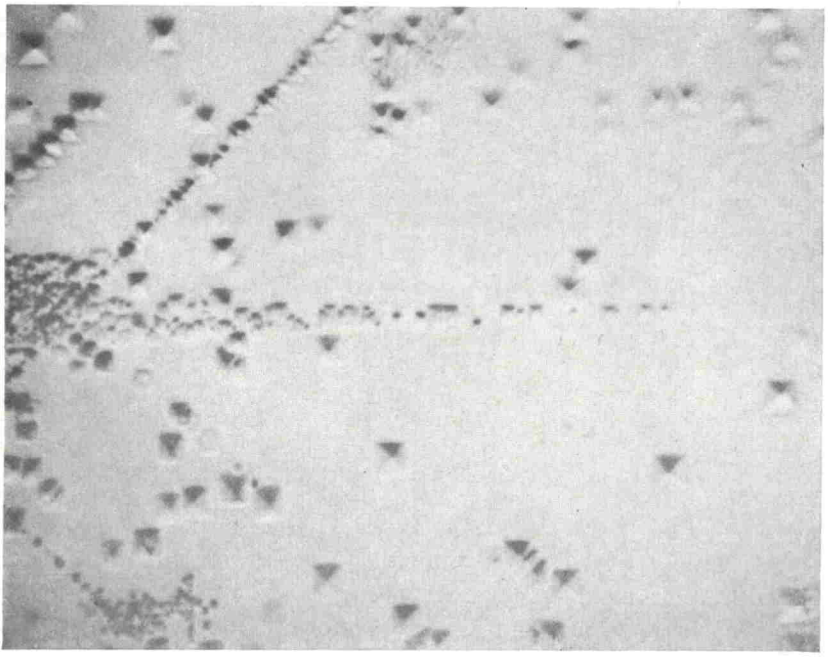

b

Fig. 1. a) Etched (100) face of a lithium fluoride crystal after stressing once at 1 bar and once at 4.3 kbar (315 ×).
b) The same region after stressing at 1 bar again and etching once more (315 ×)

handling the crystal. Dislocation velocities were determined in lithium fluoride at each given stress by averaging between 20 and 150 pit displacements such as those seen in Fig. 1; in potassium chloride average displacements of the lead dislocation in linear arrays of edge dislocations were measured.

The corrected dislocation velocity versus stress results (see Appendix) of Haworth et al. for type A lithium fluoride are given in Fig. 2. Results for type B lithium fluoride and for potassium chloride are presented in Fig. 3 and 4 respectively. There A, B refer to different impurity concentrations (see Table 1).

The results of representative macroscopic tests on lithium fluoride and potassium chloride respectively are shown in Fig. 5 and 6 (compression) and Fig. 7 and 8 (bending). The apparent difference in the intial work hardening rate between compression and bending tests (compare Fig. 5 and 7, for example) arises solely from the non-uniform stress distribution during bending, and not from any variation in dislocation behaviour between the two cases [5].

The dependence of the measured mean dislocation velocity $\bar{v}$ on the applied shear stress τ is described by either of the usual empirical equations:

$$\bar{v} = v_0 \exp\left(-\frac{D}{\tau}\right), \tag{1}$$

where v_0 and D (the so-called "drag stress") are constant; or

$$\bar{v} = v_1 \left(\frac{\tau}{\tau_1}\right)^m, \tag{2}$$

where τ_1 is the stress necessary to drive dislocations at a velocity $v_1 = 10^{-3}$ cm/s, and m is constant. Fig. 2, 3, and 4 correspond to equation (1). As shown in

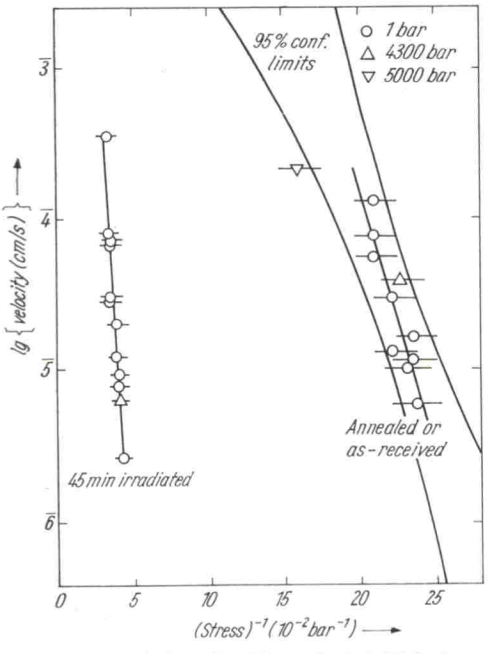

Fig. 2. Corrected results of Haworth et al. [2] for type A lithium fluoride crystals. Screw dislocation velocity vs. reciprocal resolved shear stress

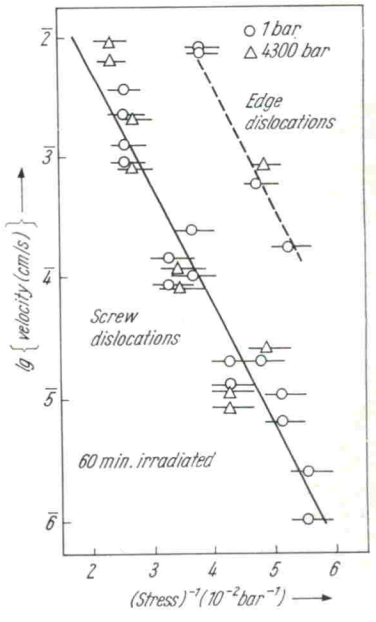

Fig. 3. Dislocation velocity vs. reciprocal resolved shear stress for type B lithium fluoride crystals

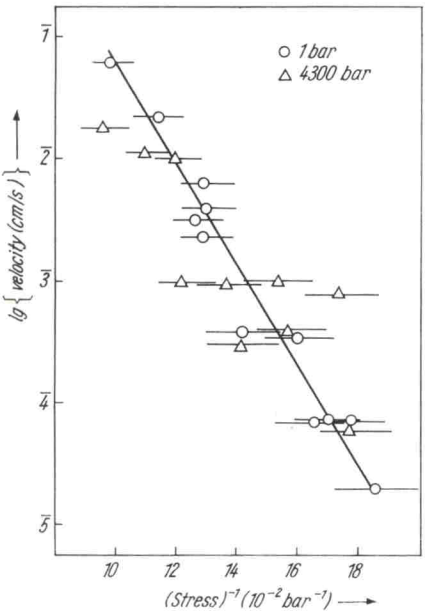

Fig. 4. Edge dislocation velocity vs. reciprocal resolved shear stress for potassium chloride crystals

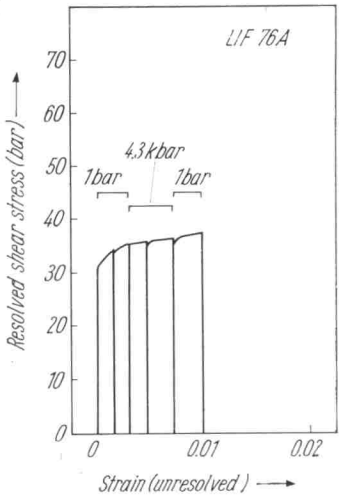

Fig. 5. Interrupted stress–strain curve for irradiated type B lithium fluoride in compression

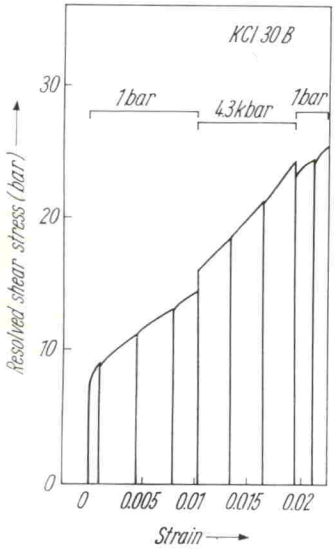

Fig. 6. Interrupted stress–strain curve for annealed potassium chloride in compression

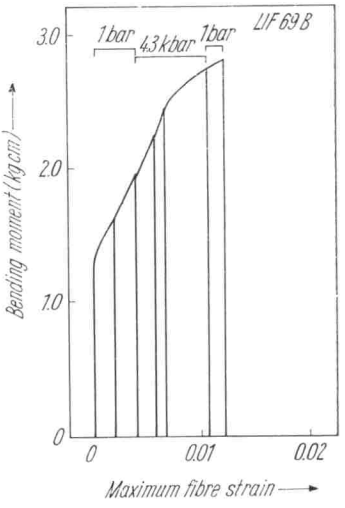

Fig. 7. Interrupted bending moment vs. strain curve for irradiated type B lithium fluoride

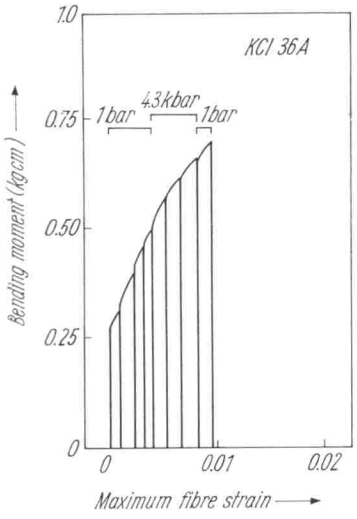

Fig. 8. Interrupted bending moment vs. strain curve for annealed potassium chloride

Table 2, the constants of (1) and (2) are unaffected by increasing the pressure on a given material: that is, increasing the hydrostatic pressure from 1 bar to 4.3 kbar has no detectable effect on the velocity–stress relationship in either lithium fluoride or potassium chloride in the range studied. The present method is sensitive enough to detect changes of more than about $\pm 5\%$ in the stress required to produce a given velocity. The macroscopic tests (Fig. 5 to 8) also exhibit no pressure dependence up to 4.3 kbar except in the case of compression tests on potassium chloride (Fig. 6) where a flow stress increase of up to 10% is observed at 4.3 kbar.

Table 2

Constants of equations (1) and (2) compared for 1 bar and 4.3 kbar. (The pre-exponential factors v_0 are accurate to no better than a factor of 10 because of the extrapolation involved in determining them.)

	LiF (type B) irradiated 60 min		KCl annealed	
Pressure	1 bar	4.3 kbar	1 bar	4.3 kbar
v_0 (cm/s)	1	1	10^3	10^2
D	1.0 ± 0.1	1.2 ± 0.2	0.42 ± 0.04	0.35 ± 0.08
τ_1 (bar)	36 ± 1	35 ± 1	7.2 ± 0.1	7.2 ± 0.1
m	9 ± 1	9 ± 2	12 ± 1	12 ± 2

3. Discussion

It can be seen by comparing Fig. 3 and 4 with Fig. 7 and 8 that the microscopic (dislocation velocity) and macroscopic experiments give mutually compatible results, as expected from the microdynamical theory, when conducted in four-point bending on similar crystals. Fleischer [6, 7] has proposed that hardening in alkali halide crystals is caused by the interaction of dislocations with the asymmetric elastic strain fields associated with interstitial atom–vacancy pairs or divalent impurity atom–vacancy pairs. This model applies in the case of irradiated crystals, and may also be valid for impure annealed crystals even when the amount of impurity present is very small [8]. The lack of pressure dependence of dislocation mobility is compatible with Fleischer's model, provided that the pressure dependence of Fleischer's "tetragonality parameter" $\Delta\varepsilon$, which describes the defect strain field, is negligible. This appears to be a reasonable assumption, although estimates of $\Delta\varepsilon$ have been made only from a greatly simplified model [9]. However, the high pressure results obtained here do not eliminate the possibility that elastic interactions between dislocations, dragging of dislocation dipoles, or even a Peierls-Nabarro mechanism, could control the dislocation mobility in these crystals. The present results do rule out a mechanism of dislocation motion that involves the formation of

cation vacancies or interstitials, in particular by the dragging of elementary jogs on screw dislocations in lithium fluoride.

Fontaine and Haasen [10] have predicted that above a critical pressure, p_c, the width of stacking faults in ionic crystals will decrease with increasing pressure, thus increasing the mobility of extended dislocations and lowering the flow stress of the crystal. For lithium fluoride p_c was estimated to be approximately 6 kbar [10]. Interrupted stress–strain tests were therefore conducted in compression on both annealed and irradiated type B lithium fluoride crystals (see Table 1) at 1 bar and 7 kbar. No change is observed in the 1% flow stress of irradiated crystals, but there is a drop of about 5% in the flow stress of annealed crystals under pressure. Extended dislocations may therefore play a significant part in the deformation of annealed lithium fluoride crystals. However, p_c for potassium chloride is about 2 kbar, and no decrease of flow stress or increase of dislocation velocity is observed at 4.3 kbar in this material.

In an earlier investigation [11, 12] the velocity–stress relationship in annealed lithium fluoride containing 100 to 200 ppm cationic impurities was found in some cases to be sensitively dependent on hydrostatic pressure, in contrast to the present results on pure crystals. Small variations in impurity content are known to cause marked changes in the dislocation mobility and flow stress of lithium fluoride [13]. Thus if the application of pressure should lower the symmetry of distortions associated with multivalent impurities whilst having no effect on distortions around irradiation-induced (interstitial) defects, this would account for the difference between the two sets of results. It is difficult to visualize such a process, and a more plausible explanation for the discrepancy arises from the differing thermal histories of the crystals used. Johnston [13] has shown that the properties of very pure lithium fluoride crystals are relatively insensitive to annealing treatment, whereas crystals containing about 80 ppm divalent impurities are harder when cooled slowly after annealing. The hardening was found to be consistent with the hypothesis that precipitates or impurity clusters form as the crystal cools, and Dryden et al. [14] have shown by dielectric loss measurements on alkali halides doped with divalent cations that impurity–vacancy pairs tend to aggregate during slow cooling. The decrease of dislocation mobility under pressure observed in impure annealed lithium fluoride crystals [11, 12] may therefore be rationalized as follows. If a moving screw dislocation is forced to cross-slip in order to pass by an impurity cluster, a jog will be formed at which point defect creation must then occur in order to allow further movement of the dislocation line. In the case of dispersed impurities or irradiation defects, the elastic interaction slows down the dislocation without necessitating jog formation, so no point defects are produced. Thus in irradiated crystals, or crystals containing only dispersed impurities, the dislocation mobility is independent of pressure. In crystals containing impurity clusters, however, point defects are produced as screw dislocations move, and so the dislocation mobility is pressure dependent [5, 11, 12]. It is necessary to assume that, predominantly, jogs of less than three or four atomic distances are produced by the cross slipping process since longer jogs lead to the formation of dipole trails rather than point defects and the dislocation mobility is then effectively unchanged under pressure. Thus if precipitates rather than submicroscopic clusters form during cooling, the subsequent dislocation velocity will not be pressure-sensitive.

We have recently conducted interrupted stress–strain tests on annealed and slowly cooled lithium fluoride crystals containing about 50 ppm cationic impurities, and observed no change in flow stress under 4.3 or 7.0 kbar pressure. However, the earlier observation [11] that a pressure effect occurs below 10 kbar in one set of doped crystals, and not in another set prepared in the same way, suggests that the effect is very sensitive to the distribution of impurities present. Further investigation is necessary to clarify the combined effects of annealing treatment and impurity concentration on dislocation mobility.

The present results do not provide strong evidence for a pressure dependence, either of dislocation velocity or of flow stress, that is directly proportional to the change in elastic constants with pressure. Such a correlation was found by Davis and Gordon [1] who observed a 25% increase in compressive flow stress for potassium chloride, containing up to 100 ppm cationic impurities, at 4.3 kbar. The present potassium chloride crystals (Table 1) exhibit only 5 to 10% increase in compressive flow stress at 4.3 kbar and less than 15% at 7.0 kbar, and little or no change is observed in bending tests or in dislocation velocity. Tests at higher pressures are necessary on pure material in order to settle this question unequivocally.

Acknowledgement

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Appendix

The absolute values of the applied stresses reported by Haworth et al. [2] should be corrected for friction in the dead weight loading apparatus and for a new load cell calibration. For the friction correction measured stresses should be reduced 20% whilst the stresses measured by means of the load cell should be reduced 25%. The corrected results are shown in Fig. 1 of the present paper. Finally, the stresses shown in Fig. 2 of the paper by Haworth et al. are unresolved fibre stresses, not resolved shear stresses as stated in the figure caption. The discussion and conclusions of the previous paper are not altered by these changes.

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