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TECHNICAL REPORT
63-13

MAY 1963

THE SURFACE ENERGY OF {100} POTASSIUM CHLORIDE.

By
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and
T. T. HITCH

FIRST TECHNICAL REPORT
OFFICE OF NAVAL RESEARCH PROJECT
NONR-4162(00)

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First Technical Report to O.N.R.

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May, 1963

Research Institute for Advanced Studies
Baltimore 12, Maryland

ABSTRACT

A modified Obreimov-Gilman cleavage technique utilizing the apparent variation of cleavage surface energy with crack length and specimen dimensions has been used to determine the true surface energy, γ_0, of the $\{100\}$ planes of potassium chloride at room temperature. γ_0 $\{100\}$ KCl was found to be 110 ± 5 ergs/cm^2, in good agreement with Born and Stern's theoretical estimate of γ_0 (108 ergs/cm^2) but not with more recent estimates of this quantity (160-185 ergs/cm^2). Possible causes for this discrepancy are discussed.

Other experiments revealed that the presence of a solvent environment at the crack tip increased the cleavage fracture energy of potassium chloride only some two or three times. This suggests that the Gurney-Pulliam phenomenon of precipitation at a crack tip is not primarily responsible for the increased strength and ductility observed when certain ionic crystals are tested in solvent environments (Joffe's - effect).

1. INTRODUCTION

The cleavage technique, as developed by Obreimov(1) and Gilman (2), provides a simple and direct means of determining the surface energy, γ_0, of the cleavage planes of certain relatively brittle materials. Under appropriate experimental conditions, γ_0 can be readily estimated to within ± 10-20%, and recently values of the cleavage surface energies of mica (1, 3), LiF $\{100\}$, CaF_2 $\{111\}$, Si $\{111\}$, Mg0 $\{100\}$ (2) (4) and Zn $\{0001\}$ (2, 5) have been obtained in this way. A similar technique also has been developed to determine the fracture surface energy of such isotropic materials as poly (methyl methacrylate)(6).

The Gilman technique involves measuring the minimum load P to propagate a previously initiated crack of length L_o in a specimen of known dimensions and elastic moduli. The surface energy γ_0 is then computed from the relationship

$$\gamma_0 = (6P^2 L_o{}^2 / E w^2 t^3) \tag{1}$$

where the dimensions L_o, w and t are as indicated in Fig. 1, and E is Young's modulus in the direction of crack propagation.

Now, since Eq. (1) is derived from elementary beam theory, it provides a reliable estimate of γ_0 only if L_o is large with respect to t(7). However, in such relatively ductile materials as potassium chloride or zinc

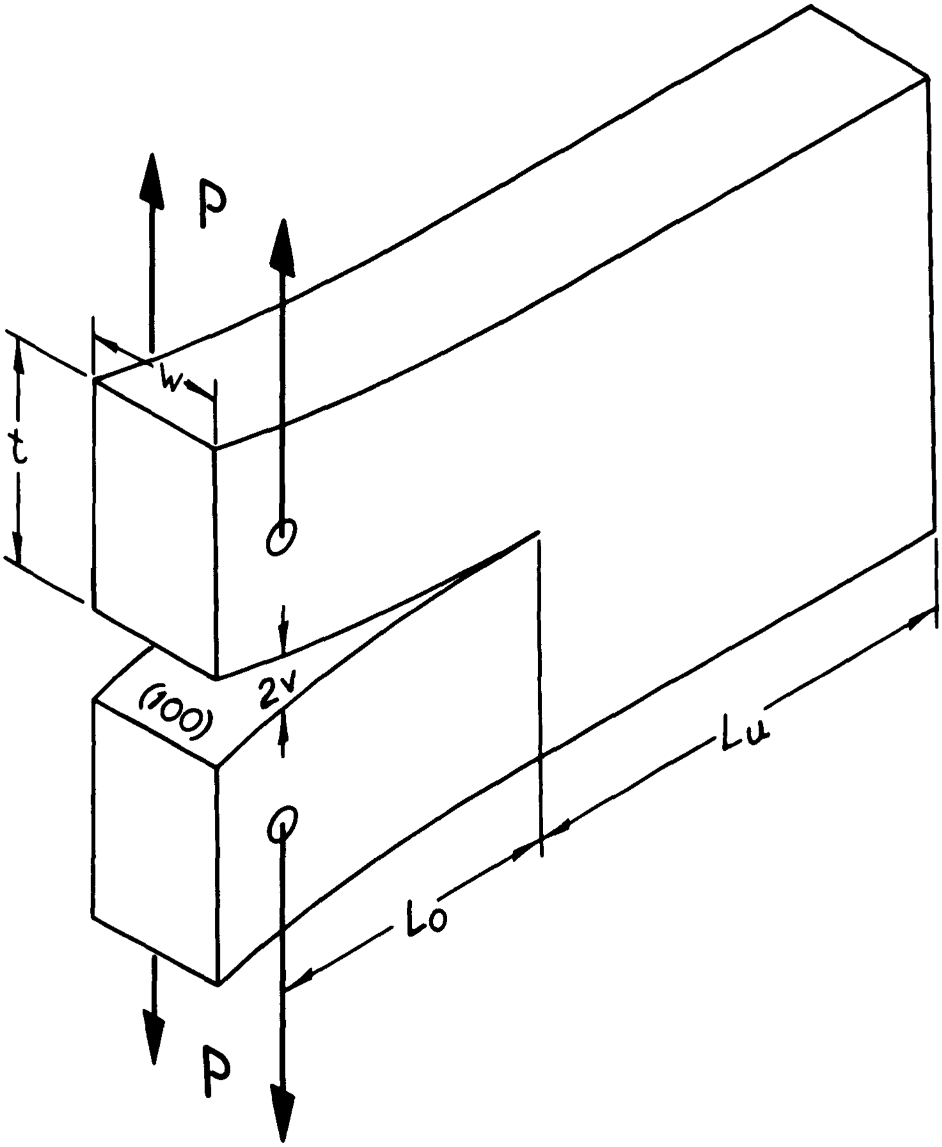

Fig. 1. Schematic of specimens used in this investigation.

monocrystals at room temperature, it is experimentally observed (5) that if L_o is greater than about 2t, then use of Eq. (1) often provides a value of γ_0 several times larger than theoretical estimates of γ_0, or of values determined by other techniques. Probably this is because the propagation of long cracks in such materials is preceded by plastic relaxation at the crack tip, and plastically blunted cracks require a greater stress to induce repropagation.

It might be expected that one way of reducing this confusing effect would be to use relatively short cracks. In this case, however, the effects of shearing forces on the deflection of the arms of the specimen, boundary conditions near the crack tip and other factors (7) neglected in the derivation of Eq. (1) become significant. As a result, the use of Eq. (1) then provides anomalously low values of γ_0.

In the present work, a modification of the Gilman approach has been adopted. Again based on elementary beam theory, but taking into consideration the contributions of shearing forces and end-condition effects(7), a relationship is derived which suggests that a more accurate estimate of the true value of γ_0 for relatively ductile materials can be obtained by analysing the apparent variation in surface energy with crack length L_o and specimen dimension t.

2. THEORY OF THE METHOD

A partially cracked crystal, such as that illustrated in Fig. 1, may be considered approximately equivalent to two built-in cantilever beams of length L_o; height t; width w; and moment of inertia $I = wt^3/12$(2, 8). If a force P is applied at a distance L_o from the built-in end, then the deflection, v, at this point is given by Castigliano's Theorem(7)

$$v = \frac{PL_o^3}{3EI} + \frac{\alpha PL_o t^2}{4GI} \qquad (2)$$

where the second term in the expression represents the effect of shearing forces on the deflection, and α is a numerical constant the value of which depends on boundary conditions in the vicinity of the crack tip. From elementary beam theory, α should lie between zero and 0.5 (7); previous studies with zinc crystals (5) suggest that, under the experimental conditions used in this work, α is likely to be < 0.1.

At the instant of propagation(2), for each arm of the specimen the work done, ΔW, in extending the crack an incremental amount ΔL_o must equal the increase in strain energy, ΔE, plus the energy of the newly created surfaces, ΔS. That is,

$$\Delta W = \Delta E + \Delta S \qquad (3)$$

Now, the elastic strain energy, E, of each deflected arm is equal to Pv/2, thus $\Delta E = (dE/dL_o) . \Delta L_o = (P\, dv/2dL_o) . \Delta L_o$. Also, since $\Delta W = (P\, dv/dL_o) . \Delta L_o$; and $\Delta S = \gamma_0 . w . \Delta L_o$, Eq. (3) can be rewritten

$$\gamma_0 . w = \frac{P}{2} \cdot \frac{dv}{dL_o} \qquad (4)$$

Differentiating Eq. (2) with respect to L_o and combining with Eq. (4) we find that

$$\gamma_0 = \frac{6P^2 L_o^{\ 2}}{Ew^2 t^3} \cdot \left(1 + \frac{\alpha E}{4G} \left(\frac{t}{L_o} \right)^2 \right) \qquad (5)$$

That is to say

$$\gamma_0 = \frac{6P^2 L_o^{\ 2}}{Ew^2 t^3} \cdot (1 + C_s)$$

where $C_s = (\alpha E t^2/4GL_o^{\ 2})$ and is the correction factor which takes into account the effects of shearing forces and end-conditions. If α is assumed to be ~ 0.1 (5), then when $L_o \geq 3t$, as in Gilman's experiments (2), C_s is < 0.01 and therefore may be neglected. However, when $L_o \leq t$, as in some of the experiments to be described, C_s becomes an extremely significant factor in the determination of a reliable value of γ_0 .

Now, from an experimentalists' point of view, equation 5 can be conveniently rewritten in the form

$$\left(\frac{1}{\dfrac{6P^2L_o^2}{Ew^2t^3}}\right) = \frac{1}{\gamma_o} + \frac{\alpha E}{4\gamma_o G}\cdot\left(\frac{t}{L_o}\right)^2 \qquad (6)$$

where the value of $(6P^2L_o^2/Ew^2t^3)$ may be regarded as an "apparent" surface energy, γ_A. Thus a plot of $(1/\gamma_A)$ versus $(t/L_o)^2$ should be linear, of intercept $(1/\gamma_o)$ at $(t/L_o)^2 = 0$, and of slope $(\alpha E/4\gamma_o G)$. This approach should, by limited extrapolation of the experimental data, enable accurate estimates to be made of γ_o and α. The resulting value of γ_o is that which would have been obtained had it been practical to use specimens containing the extremely long cracks required by elementary beam theory.

3. EXPERIMENTAL

In this work, the variation of $(1/\gamma_A)$ with $(t/L_o)^2$ for potassium chloride crystals was studied at room temperature. Monocrystal blocks of potassium chloride were obtained from the Harshaw Chemical Company, and specimens approximately 30 mm x 2-4 mm (w) x 6-20 mm (2t) cleaved from these, Fig. 1. Cleavage cracks were introduced either by means of a crack-initiating jig similar to that described in refs. 2 and 5, or with a hand-held, hollow-ground chisel in the normal way. Cracks were prevented from propagating completely through the specimens by the application of small compressive stress perpendicular to the direction of propagation (2).

To be considered suitable for subsequent testing, initiated cracks were required to exhibit (i) relatively straight "fronts" lying perpendicular to the direction of propagation, (ii) widely spaced interference fringes near the crack tip, and (iii) be of such geometry that $L_o < L_u$, and $L_u > t$, Fig. 1. Even with practice, not more than one in three of the initiated cracks met all these requirements. Nevertheless, this selection was found to be essential in order to obtain self-consistent data.

Specimens containing suitable cracks were promptly mounted in the propagation rig, Fig. 2, attached to the crosshead of an Instron testing machine, taking care to ensure that the cleavage plane was accurately perpendicular to the loading axis. Use of the alignment jig, Fig. 2, prevented accidental damage to the specimen during mounting. The crack length, L_o, Fig. 1, was then set at an appropriate value, ranging from 2-15 mm, so as to provide a predetermined value of the ratio $(t/L_o)^2$, and the rate of loading used was $\sim$ 20 g/sec. The load P to repropagate the crack, indicated by a sharp drop in the recorded load deflection curve, was noted.

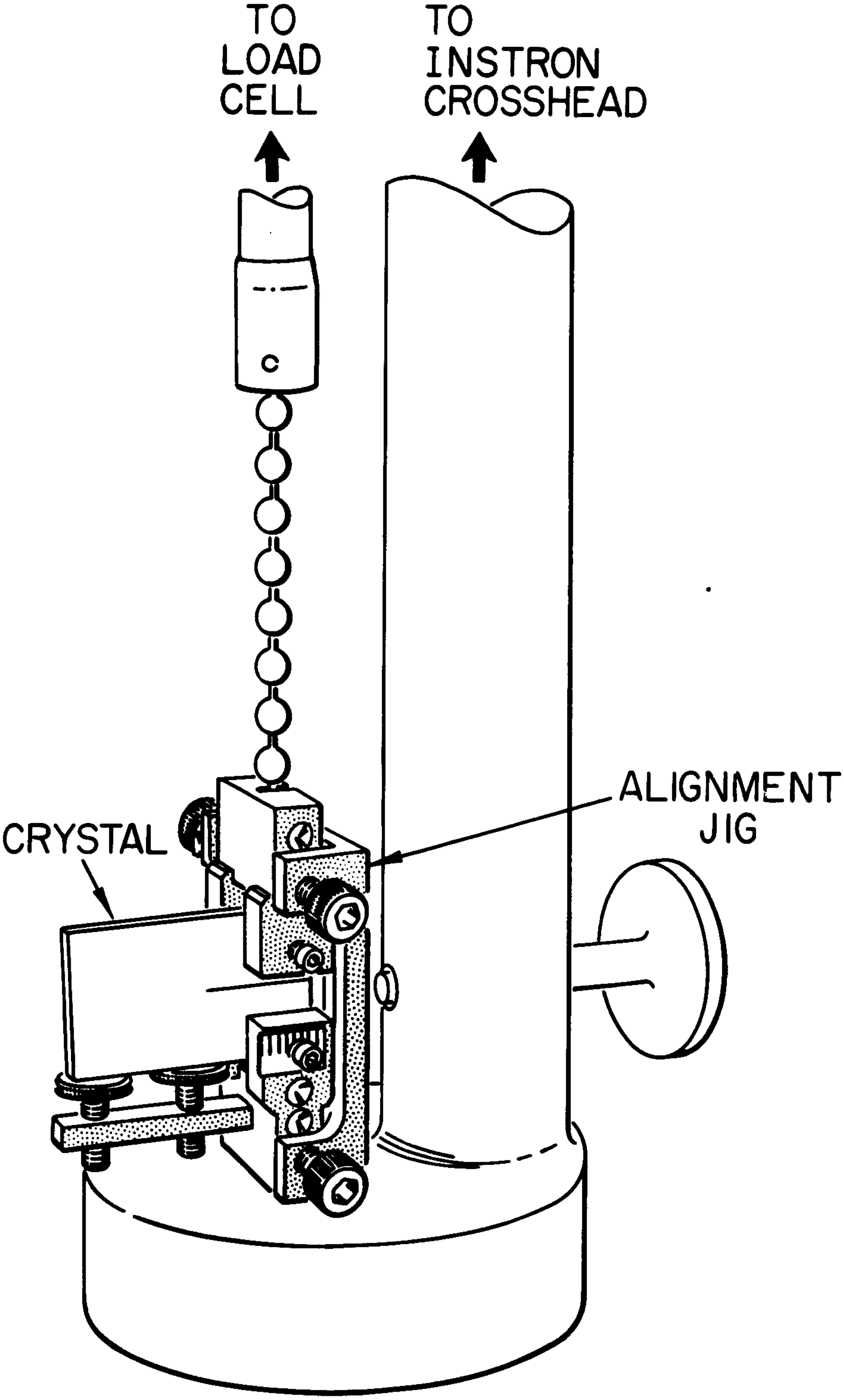

Fig. 2. Crack propagating rig with crystal and alignment jig in position.

Crack fronts revealing a high density of cleavage steps emanating from any portion of the front were rejected from further consideration. For those specimens surviving this final selection, values of L_o, w and t were obtained optically to within ± 0.02 mm.

Values of the elastic moduli taken from the work of Durand(9) were used to compute the "apparent" surface energy γ_A and this function was then plotted against $(t/L_o)^2$.

Other experiments were performed to investigate the possibly significant effects of solvent environments on crack propagatibility(10-12). To this end, crack tips were exposed either to water (1-2 min) or to a saturated aqueous solution of KCl (5-8 min) prior to testing. Two techniques were employed; either distilled water was supplied to the crack tip by means of a micropippette, or specimens were totally immersed in the saturated salt solution. Now, according to Pulliam, when either of these environments is fed into the crack, precipitation of KCl occurs specifically at the crack tip - not on the crack walls. After an exposure of about 1 min, the length of the crack should decrease by ∼ 0.012 mm, and the tip radius, r, should increase from less than 10 A to about 2000 A. This increase in r might be expected to have a pronounced effect on the load **P** to propagate the crack, since it is well known that **P** is proportional to $r^{1/2}$ (13).

4. OBSERVATIONS AND DISCUSSIONS

4.1. Determination of γ_0 for $\{100\}$ KCl.

The variation of $(1/\gamma_A)$ with $(t/L_o)^2$ for potassium chloride monocrystals at 298°K is illustrated in Fig. 3. Data points are scattered within a band which converges as $(t/L_o)^2$ approaches zero. The line drawn is considered to be a best fit to the data and intersects the ordinate axis at $(1/\gamma_0) = 0.0091 \pm 0.0002$ cm^2/erg. The surface energy of the $\{100\}$ faces of KCl, therefore, is determined to be 110 $\pm$ ($<$5) ergs/cm^2 and the constant α to be 0.043.

The data represented by open circles at $(t/L_o)^2 < 0.4$ illustrate the manner in which tests performed with relatively long cracks lead to anomalously high values of γ_A and hence of γ_0 (4, 5). Values of γ_A for cracks of $(t/L_o)^2 < 0.4$ ranged from 114 ergs/cm^2 to 149 ergs/cm^2.

The divergence of the scatter band with increasing $(t/L_o)^2$ is associated (i) with the increased importance of errors in the measurement of L_o when L_o is small ($\sim$ 2 mm) since crack fronts are always somewhat rounded, (ii) with the plotting of the reciprocal of γ_A as ordinate, and (iii) with the increasing inapplicability of elementary beam theory as L_o decreases with respect to t. Indeed, the limited deviation from linearity over the range of $(t/L_o)^2$ employed is quite remarkable.

It is of interest to compare the value of $\gamma_0\{100\}$ KCl determined in the present work with previous theoretical and experimental estimates of this quantity. Table I summarizes this data. The present value of

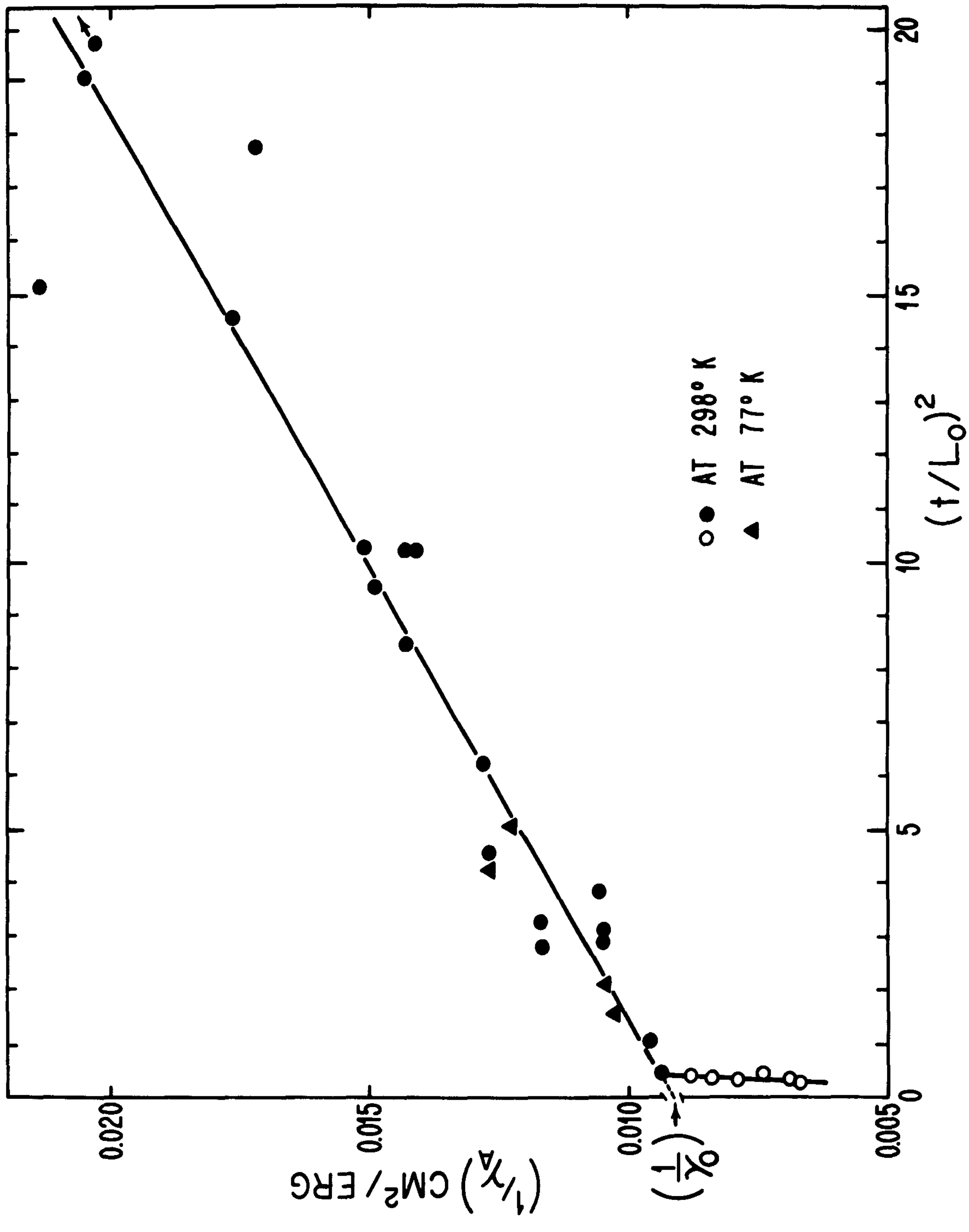

Fig. 3. Effect of variations in crack length L_o and specimen dimension t on the apparent cleavage surface energy γ_A of {100} KCl at 298°K.

110 ergs/cm^2 should be compared only with those estimates of γ_0 for unrelaxed, non-polarized surfaces. Since the polarization and relaxation process occurs after the fresh surfaces have been created by the passage of the crack, the decrease in surface energy associated with this phenomenon will not be recorded by the cleavage technique. Table I reveals that the estimate of Born and Stern (14) most closely approximates the value determined in this work. Yet these workers utilized the simplest of atomic models as a basis for their calculations - the electrostatic attractions of unlike ions and the repulsions of hard shells. More recent theoretical studies (19-21), however, have also taken into account the contributions of Van der Waals forces and other factors; these considerations have led to estimates of γ_0 of 160 to 185 ergs/cm^2.

In recent years, the early estimates of Born (14), Lennard-Jones (15) and others have been regarded as of doubtful value because (i) they neglected the contributions of Van der Waals forces, and (ii) they were approximately one half the values of γ_0 determined experimentally from surface tension studies with molten salts (23) and by calorimetric studies (24). Now, while the first criticism is certainly valid, it might well be considered that the estimates of γ_0 from such experiments are not above suspicion. For example, the value of γ_0 at 0°K (173 erg/cm^2) deduced by Jaeger (23) involved extrapolation over a wide temperature range and did not allow for the obvious differences in molecular arrangement in solids and liquids, especially at the surface. Furthermore, the value determined by Balk and Benson (24)

TABLE I. Summary of Data on Surface Energy of {100} Face of Potassium Chloride (ergs/cm^2).

Author	Reference	Method	Value
THEORETICAL ESTIMATES			
Born & Stern (1919)	14	Elementary electrostatic approach	108
Lennard-Jones & Taylor (1925)	15	As above	77
Dent (1929)	16	Similar to Born but allowing for surface polarization	(56)*
Biemuller (1936)	17	As above	(91)
Glauberman (1949)	18	As above	107(91)
Shuttleworth (1949)	19	Using complete Born-Mayer equation	163(134)
Van Zeggeren & Benson (1956)	20	Quantum Mechanical approach	184
Van Zeggeren & Benson (1957)	21	Similar to Shuttleworth	163
Benson (1961)	22	As above	175(145)
EXPERIMENTAL DETERMINATIONS			
Jaeger (1917)	23	Extrapolation of Data from Molten Salt Experiments	173(at 0°K)
Balk & Benson (1959)	24	Heat of Solution (Surface Enthalpy of Smokes)	252(at 298°K)
Present work		Cleavage Technique	110(at 298°K)

* Values in brackets take into consideration ionic polarization and relaxation at the free surface.

(252 ergs/cm^2) - using a heat of solution method and potassium chloride powder approximately spherical in shape - must be considerably greater than the true $\{100\}$ since, in fact, it represents an average surface energy of many crystallographic planes. (All planes other than $\{100\}$ are of higher surface energy than $\{100\}$).

The better agreement of the simpler theories with experimental data produced by the cleavage technique has also been noted by Gilman(2). Assuming the data produced by this technique to be reliable at least to within ± 10%, then since the principal difference between early and recent theoretical estimates of γ_0 appears to lie in the estimated contribution of Van der Waals forces, one wonders whether the relative significance of the binding due to these forces may not have been overestimated for simple ionic crystals. Alternatively, Benson and Dempsey(25) have noted that while theoretical estimates of the cohesive energy are not particular sensitive to slight variations in ionic radii, surface energy calculations are significantly affected because such variations markedly affect the calculation of repulsive energies. They comment that perhaps a different approach to the surface energy calculation is required, preferably one which makes use of empirical surface properties of the crystal as well as volume properties.

4.2 Effects of Solvent Environments on Crack Propagation in Potassium Chloride-Joffe's Effect.

A marked increase in strength and ductility is often observed when crystalline materials are tested in a solvent environment. There are several causes for such behavior (26) including, for example, the removal, by dissolution, of surface sources of dislocations or of embrittling surface films. However, for ionic crystals, one of the most important causes of the phenomenon is the effective elimination of pre-existing surface cracks. Joffe et al (27) considered that the complete removal of such surface defects by dissolution was necessary before the significant improvement in mechanical properties (strength increased some 25 times, ductility perhaps 10 times) could be observed. More recently, however, Gurney (11) and Pulliam (12) have suggested that a solvent environment might markedly increase ductility and strength simply by reducing the propagatibility of existing cracks. As mentioned in section 3, crystal dissolution followed by reprecipitation at the crack should decrease the notch-embrittling action of the crack by increasing the radius of curvature of its tip. To examine the effects of reprecipitation on crack propagatibility, specimens were tested following exposure of the crack tip to water and saturated solutions of KCl. The data is presented in Fig. 4.

In Fig. 4, the intercept at $(t/L_o)^2 = 0$ defines a fracture energy, ϕ_0, rather than a true surface energy. Thus ϕ_0, where $\phi_A = (6P^2L_o^2/Ew^2t^3)$, is the fracture energy to propagate a crack in a solvent environment. Though there is much scatter in the data, it is apparent that ϕ_0 is about 215 ± 20 ergs/cm^2. Now since ϕ_0 is only about $2\gamma_0$, and since, on the Griffith criterion, the fracture stress is proportional to the square root of the fracture (surface) energy, it seems unlikely that the Gurney-Pulliam precipitation mechanism

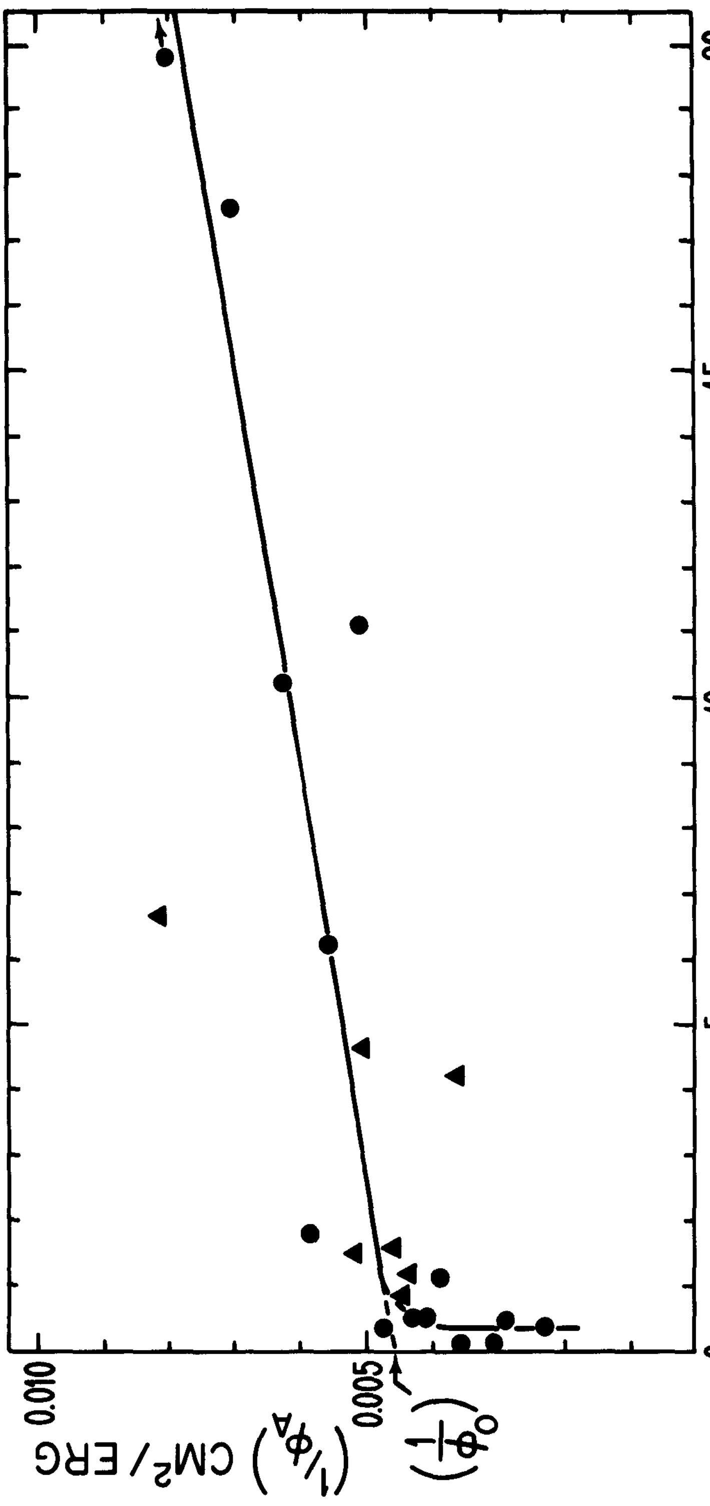

Fig. 4. Effect of solvent environments at the crack tip on the cleavage fracture energy ϕ_A for {100} KCl at 298°K.
Exposed to water for 1-2 min.
Exposed to saturated KCl solution for 5-8 min.

can be primarily responsible for the large increases in tensile strength observed when ionic crystals are tested in solvent environments (25). It appears more likely that Joffe's original explanation (involving the complete removal of surface defects) is correct.

However, there is also a discrepancy between the small increase in effort required to propagate cracks following exposure to solvent environments observed in this work, and that expected on the basis of the large increase in crack tip radius resulting from similar exposures reported by Pulliam. Since the energy required for crack propagation is proportional to the radius of the crack tip(5), the ratio ϕ_0/γ_0 might have been expected to be of order several hundred instead of two to three. However, for this to be observed, the precipitate at the crack tip would have to be completely coherent with the specimen lattice so that a perfect, single crystal "weld" resulted. The small ratio of ϕ_0/γ_0 reported here suggests that instead the precipitate formed was an incoherent, polycrystalline mass of low tensile strength.

4.3 Effects of Irradiation on $\gamma_0\{100\}$ KCl.

A few specimens of potassium chloride were irradiated with gamma rays from a cobalt 60 source (2×10^6R. - 10^8R.) prior to crack initiation. It was considered that this treatment might effectively reduce the extent of plastic relaxation at the crack tip as a result of dislocation-point defect interactions(28), yet not significantly affect surface energy determinations because of the relatively small density of defects introduced(2). However, it proved impossible to obtain reproducible data with these irradiated specimens because of the persistent (and sometimes spontaneous) crack-healing or coalescence phenomena discussed recently by Shaskol'skaya et al (29).

5. SUMMARY

(1) A new approach to the cleavage technique has been described which permits more accurate determinations of γ_o for certain materials than hitherto.

(2) The surface energy of $\{100\}$ KCl has been determined to be 110 ± 5 ergs/cm^2 at 298°K.

(3) Studies of crack propagation in solvent environments revealed that the Gurney-Pulliam phenomenon of precipitation at a crack tip probably is not primarily responsible for Joffe-effect's in ionic crystals.

ACKNOWLEDGEMENTS

It is a pleasure to acknowledge the useful discussions with Dr. J. K. Mackenzie and the capable assistance of D. L. Goldheim and H. Opperhauser in this work. We also appreciate the financial support received from the Office of Naval Research during the latter part of the investigation.

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