MECHANICAL PROPERTIES OF POTASSIUM CHLORIDE

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CHAPTER I

INTRODUCTION

The current interest in alkali halides, KCl in particualr, is sparked by a desire to use them as window materials for high power CO₂ lasers. KCl has several advantages that make it appealing for this application. One advantage is that it is both readily available and relatively inexpensive. Another advantage of greater importance is its low infrared optical absorption. Pure KCl has a serious disadvantage in that it exhibits a low mechanical strength.

The problem, then, is how to increase this mechanical strength without distrubing its infrared absorption properties. The first hint of a solution to this problem was discovered by Goldstein (1) late in the 1800's. This occurred when Goldstein created a Farbzentren or F center in some alkali halide samples by bombarding them with an electron beam. The same effect was noted later when the irradiation was done with X-rays, and later still with gamma rays. It has been shown by Nadeau (2,3) and by Sibley and Sonder (4) that this type of F center creation produces significant hardening of alkali halide samples. In 1932 Edner (5), Metag (6), and Schönfeld (7) showed that the flow stress of sodium chloride increased when small concentrations of divalent cations were grown into the crystal lattice. Both of these hardening methods harden the sample by creating tetragonal lattice distortions. Radiation causes this distortion by converting a negative ion to a neutral atom and moving the atom to an interstitial position, leaving the electron in the original position, forming an F center. Doping with divalent ions creates distortion, because for the sample to remain electrically neutral a positive ion vacancy must be created. This vacancy pairs with the two plus ion creating a tetragonal defect. Fleischer (8) has developed a theory to explain the hardening of face centered cubic crystals by tetragonal lattice distortions.

The degree of hardening obtained by the doping of KCl with Ca⁺⁺ and Sr⁺⁺, by irradiating pure KCl, and by forming mixed crystals of $KCl_{x}KBr_{1-x}$ as compared to the initial hardness of pure KCl, pure KBr, and pure NaCl was measured using several techniques. The list of techniques employed includes the Vickers microhardness test, flow stress determination by both uniaxial compression and four point bending tests, and measurement of the size of the dislocation rosette around a Vickers indentation. The increase in hardness observed will be compared to predictions made by Fleischer's theory.

CHAPTER II

EXPERIMENTAL METHOD

The KCl samples were cleaved from Czochralski grown crystal boules. These samples were grown at Oak Ridge National Laboratory. Their characterization has been given by Sibley and Russell (16). All samples used in this work are from 8 to 10 years old; therefore, the results from these samples may not be typical of those for freshly grown crystals since some precipitation of the dopants may have occurred. The samples of KBr and NaCl used in this work were obtained from Harshaw.

The KCl samples used in determining the effect of radiation damage upon the resolved flow stress were irradiated by a 10 µA current of 1.5 MeV electrons for various lengths of time in the Oklahoma State University Van de Graaff facility. Since this type of irradiation creates interstitials and F centers simultaneously, the number of F centers is a convenient measure of the amount of radiation damage. The F center concentration was determined from absorption spectra obtained on a Cary 14 spectrophotometer using Smakula's formula (9,10). Smakula's formula yields the F center concentration N_F (cm⁻¹) when applied to a Gaussian shaped absorption peak, and is given by

$$N_{\rm F} f = 0.87 \times 10^{17} N_{\rm o} \alpha_{\rm max} W_{\rm 1} / (N_{\rm o}^2 + 2)^2$$
 (1)

where f is the oscillator strength, $W_{l_{k}}$ (eV) is the width at half

maximum of the F band peak, N is the index of refraction, and α_{max} is the F band absorption coefficient.

The strength of the samples was measured using four different techniques. They are the uniaxial compression test, the four point bend test, the Vickers microhardness test, and the wing size test. Each of these techniques is an independent method for determining the mechanical strength of a sample. Except for the Vickers microhardness test, they either give the flow stress directly, or give a value that is inversely proportional to the flow stress.

The uniaxial compression test was run on an Instron testing machine which recorded the force applied to the sample by means of a load cell and chart recorder while the sample was compressed at a constant rate of strain. The flow stress was determined at a strain rate of approximately 10^{-3} sec^{-1} . The flow stress, τ_0 , is taken as the stress value at the point of intersection of tangents drawn to the elastic and first plastic regions of the stress strain curve as shown in Figure 1. τ_0 is calculated by taking the force at the point specified above and dividing by the cross sectional area A of the sample. The resolved flow stress, τ_r , which equals the shear stress, is calculated by dividing the force component parallel to the slip plane by the area of the slip plane as shown in Figure 2. The resolved . flow stress can be written as

$$\tau_{r} = F \sin \phi \cos \phi / A$$
$$= \tau_{sin} \phi \cos \phi \qquad (2)$$

where F = applied force, A = the cross sectional area of the sample, $and <math>\phi$ is the angle the slip plane makes with the central axis along









which F is being applied. For KCl, the primary slip direction is 110. The force was applied along a 100 direction; thus $\phi = 45^{\circ}$; and τ_r can be written as

$$\tau_r = \tau_0/2$$
 (3)

The four point bend test was also run on the Instron testing machine. To obtain pure bending, a special jig had to be made as shown in Figure 3. To calculate the flow stress of a sample bent by this jig, the internal bending moment is first calculated. Then, the external bending moment is set equal to it. This assumes an equilibrium state between the bending moments of the sample. To make this calculation, one assumes that the sample is in the shape of a beam with a width W and thickness t, that a length 1 exists between the support points, and that d is the distance between the force point and its associated support as shown in Figure 3. One also assumes that the sample has been bent by a total force P such that it has a radius of curvature R, but is still within its elastic limit. With these conditions satisfied, the internal bending moment M equals the integral of the incremental bending moment, Δm , over the entire cross section of the sample. Am for an incremental area, AA, a distance y from the neutral axis caused by a stress σ is given by

$$\Delta \mathbf{m} = \sigma \mathbf{y} \Delta \mathbf{A} = \sigma \mathbf{y} \mathbf{W} \mathbf{d} \mathbf{y}$$

Then

$$m = Area \Delta m = \int_{-t/2}^{t/2} \sigma y W dy . \qquad (4)$$

When y = t/2, the stress σ is equal to the maximum value it can have, σ_{max} , under the applied force P. So σ can be rewritten as



Figure 3. Diagram for a Four Point Bending Jig

 σ_{max} y/(t/2). Substituting this into the integral equation for m yields

$$m = \sigma_{max} / (t/2) \int_{-t/2}^{t/2} y^2 W dy = \sigma_{max} I / (t/2) , \qquad (5)$$

where I is the moment of inertia. For the beam, I can be evaluated as

$$I = \int_{-t/2}^{t/2} y^2 W dy = W t^3 / 12 , \qquad (6)$$

and m can be evaluated as

$$m = \sigma_{max} t^2 W/6 . \tag{7}$$

This internal bending moment was created by applying an external bending moment m'. The two bending moments must be equal since the system is in an equilibrium state. The external bending moment is equal to the applied force, P/2, times the moment arm, d. Using m' = m, the following relation is derived:

$$m' = Pd/2 = m = \sigma_{max} Wt^2/6$$
. (8)

Solving this equation for σ_{max} yields

$$\sigma_{\text{max}} = 3Pd/Wt^2 .$$
 (9)

If P is evaluated as the force at the intersection of the tangents, as in the compression test, then $\sigma_{max} = \tau_{o}$ and $\frac{1}{2}\tau_{o} = \tau_{r}$, yielding the resolved flow stress from the four point bend test.

The Vickers microhardness test is an indentation test. A square base pyramidal diamond of large apex angle is lowered onto the sample at a controlled rate of descent and under a specific dead weight load as shown in Figure 4. The hardness number that results from this type of test is a measure of the resistance of the plastically deformed



(a) Vickers Indentor



- (b) Vickers Indentation and Minimum Sample Size for a Valid Test
- Figure 4. Vickers Microhardness Indentor and Indentation Diagrams

sample to further plastic flow. That is, the indentor will continue into the sample until the sample's resistance is equal to the force applied by the indentor. The hardness number is calculated from the indentor load and the surface area of the indentation by the formula

$$HV = P/A , \qquad (10)$$

where HV = the Vickers hardness number in kg/mm^2 ,

P = the indentor load in kilograms, and

A = surface area of the indentation in mm^2 .

The surface area of the indentation is calculated from the apex angles of the diamond and the microscopically measured diagonals of the indentation. For the diamond used in this work, the formula for Vickers hardness numbers can be written as

$$HV = 1854.4 \cdot P/d^2 , \qquad (11)$$

where d is the length of the diagonals in μm .

The wing size test measures the size of the dislocation rosette around a Vickers indentation. These rosettes were revealed by etching the sample, rinsing in acetone, and blotting dry. Glacial acetic acid was found to be the best etchant for pure samples, and a saturated solution of BaBr₂ in absolute methanol was best for doped samples. The indenting and etching were carried out in a dry box with the relative humidity kept below 40%. Hopkins, Miller, and Martin (11) gave a physical description of what happens when the diamond of the indentor contacts the surface of the sample. In Figure 4a it is clear that the stress, given by the force/area, is a maximum at first contact. When this contact is made, the point on the indentor generates a string of dislocations. The first of these is pushed out by succeeding ones to the distance W_0 for which the stress applied by the indentor equals the minimum stress τ_r required to move new dislocations along their primary slip direction of <10. Therefore, the greater τ_r , the smaller W_0 . As the indentor continues into the surface, it will physically displace crystal material. If one assumes that the distance of this displacement is ΔW , then the wing size, W, measured from photographs such as that in Figure 5 will be equal to $W_0 \stackrel{*}{\rightarrow} \Delta W$. Since W_0 is the wing size that is related to τ_r , a correction for this must be made when using this test.



Figure 5. A Dislocation Rosette of a 50 gm Load Indentation on KCl:Sr

CHAPTER III

RESULTS

Table I compares the results of the four different techniques for measuring the hardness of a sample on a number of different samples. It should be noted that the units used for stress are kg/mm², rather than force/mm², and that the concentration given in ppm is a mole fraction concentration. Included in this list of samples is a series of pure and doped KCl samples for which the dopant level of Sr was varied from less than 1 ppm to approximately 470 ppm. When the resolved flow stress, τ_r (4 pt.), as determined by the four point bend test, is compared to the resolved flow stress, τ_r (uni), as determined by the uniaxial compression test, a linear relationship is obtained. This is shown in Figure 6. Since both measuring techniques yield essentially the same result for any particular measurement and the internal stress in the bend test is nonuniform, only the flow stress determined from uniaxial compression measurements will be considered further.

The resolved flow stress and Vickers hardness numbers for pure KCl are given as a function of irradiation in Table II. Figure 7 (upper curve) is a graph of this data and clearly shows the nonlinear dependence of the resolved flow stress on the radiation damage as measured by the F center concentration. The lower curve in the same figure presents the unpublished results of Sibley (12) and shows a similar but less pronounced dependence of the resolved flow stress of unirradiated doped KCl on the dopant level of Sr. For equivalent concentrations, radiation damage is more effective for hardening KCl than is Sr doping.

TABLE I

RESOLVED FLOW STRESS UNIAXIAL, τ (uni), RESOLVED FLOW STRESS FOUR POINT BEND, τ (4 pt.), VICKERS HARDNESS, HV, WING SIZE, w, AND CONCEN-TRATION OF BOPANT LEVEL, c, FOR SELECTED ALKALI HALIDES

Material	rial c* τ _r (uni) (ppm) (kg/mm ²)		τ _r (4 pt.) (kg/mm ²)	HV (kg/mm ²)	wo (µm)	
KC1-P-1	0	0.11		9.2	91	
KC1-P-2	0	0.15		9.0	91	
KC1-P-3	0	0.13	0.15	10.2		
KC1-Sr-1	50-150	0.23	0.27	13.0	77	
KC1-Sr-2	470	0.56	0.64	16.9	41	
KBr	0	0.10	0.10	7.7		
NaCl	0	0.24	0.21	16.5	6.00	

* mole fraction

TABLE	II
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RESOLVED FLOW STRESS, τ_r , VICKERS HARDNESS, HV, AND F CENTER CONCENTRATIONS, c, FOR PURE KC1

			· · · · · · · · · · · · · · · · · · ·
Material	c* (ppm)	(kg/mm^2)	HV (kg/mm ²)
KC1-P-1	0	0.11	9.2
KC1-P-2	0	0.15	9.0
KC1-P-3	0	0.13	10.2
KC1-P-3	7.1	0.43	11.4
KC1-P-3	19.4	0.64	12.6
KC1-P-3	54.0	0.85	13.2
KC1-P-3	56.3	0.87	12.0
KC1-P-3	71.3	0.93	14.8
			,

* mole fraction

ŧ









CHAPTER IV

DISCUSSION

The strengthening of alkali halide crystals can be accomplished in several ways. Two of these are hardening by irradiation and hardening by doping with divalent cationic impurities. These methods harden the sample by creating tetragonal lattice distortions. Irradiation of the sample by ionizing radiation creates a tetragonal lattice distortion by converting a negative ion to a neutral atom and moving the atom to an interstitial site, leaving the electron in the original site creating an F center. Therefore, measuring the concentration of F centers is a convenient method of determining the number of interstitials that have been formed during any particular irradiation process. That interstitials and not F centers cause the observed hardening has been established by Sibley and Sonder (4), and by Hopkins (13), who has additively and electrolytically colored pure KC1. Hopkins measured the flow stress before and after the coloring process and found no significant change. Doping the crystal with divalent ions creates tetragonal distortion because a positive ion vacancy must be created for the sample to remain electrolytically neutral. The divalent ion and the positive ion vacancy pair up to form the tetragonal defect. The axis of the tetragonal defect caused by interstitials is a $\triangleleft 0 \bigcirc$ axis while that caused by divacancies is a 🗸 1 🗘 axis.

Fleischer (8) developed a theory to explain the hardening of face centered cubic crystals by tetragonal lattice distortions. Because of the importance of this theory in explaining the hardening observed in these samples a brief review of it is in order. To compute the hardening of a crystal by defects, in general one must first compute the interaction energy of the dislocation with the defect with which it will interact. This computation is simplified if two conditions are satisfied. First the force on a dislocation due to a defect must fall off rapidly as their distance of separation increases, and second, the concentration, of these defects must be small when compared to the number of lattice sites. The second condition is usually satisfied since the solubility of defects that strongly distort the lattice is low. Fleischer demonstrated that the force of interaction falls off as the square of the distance of separation, which satisfies condition one. The calculation, then, is for a dislocation interacting only with widely scattered defects on its slip plane.

To find the minimum force necessary to move a dislocation along a slip direction one must first calculate the maximum force the crystal can exert upon the dislocation. To do this, one assumes that F_{max} is the maximum force any one defect can exert upon the dislocation and 1 is the spacing of defects along the dislocation line. Then, the minimum stress necessary to move the dislocation can be expressed as

$$\tau = F_{\max} / b1 , \qquad (12)$$

where b is the slip vector (Burgers vector) of the dislocation.

The problem has now been separated into two manageable parts, which are to find 1 and to find F_{max} . 1 is easy enough to find if the

assumption of random distribution of defects is valid. With this assumption, and by knowing the concentration of interacting defects and their cross sectional area, 1 can be calculated as

$$L = (area/concentration)^{\frac{1}{2}} .$$
 (13)

To calculate F_{max} , Fleischer first calculated the interaction energies of the dislocation with various orientations of a tetragonal defect. If the energy density E_d , which is given by the stress tensor of the dislocation times the strain tensor of the defect, is known for each orientation, then the interaction energy E is given by

$$E = E_{d}V, \qquad (14)$$

where V is the volume of the defect. In face centered cubic crystals, the atomic volume is equal to $b^3/\sqrt{2}$, so the interaction energy per atomic volume can be written as

$$E_{v} = \varepsilon_{ij} \tau_{ij} b^{3} / \sqrt{2} , \qquad (15)$$

where ϵ_{ij} is the strain tensor, τ_{ij} is the stress tensor, and repeated indices indicate summation. The total interaction energy can then be written as

$$E = nE_{u}, \qquad (16)$$

where n is the number of atomic volumes the defect occupies. For interstitials and divacancies, this number is 2.

To calculate the stress and strain tensors for a particular orientation, three assumptions are made: First, the stress from a dislocation does not vary over the volume of the defect; second, this stress is not altered by the presence of the defect; and third, isotropic elasticity can be employed. With these assumptions and the appropriate mathematical steps, Fleischer calculates the interaction energy per atomic volume between a screw dislocation that is along a [101] axis moving in the [121] direction in the (111) plane and a tetragonal defect which has a [100] axis as

$$E_{v} = -Gb^{4}\Delta\varepsilon \cos \varepsilon / (2\sqrt{2} \pi r) , \qquad (17)$$

where: f is the angle between the [010] and a line from the dislocation to the defect, and $\Delta \epsilon$ is a measure of the strength of tetragonality as shown in Figure 8. Similar calculations for a $\langle 10 \rangle$ defect yield an energy of

$$E_{v} = -Gb^{4}\Delta\varepsilon (\sqrt{2}\cos\ell + \sin\ell)/(4\pi r) . \qquad (18)$$

If f is expressed in terms of x, the distance along the slip plane, and y, the distance of closest approach, as shown in Figure 8, the energy of the $\langle 100 \rangle$ defect can be written as

$$E_{v} = Gb^{4} \Delta \varepsilon (y + \sqrt{2} x) / 2\pi \sqrt{6} (x^{2} + y^{2}) .$$
 (19)

The maximum force felt from this defect by the dislocation can be found by maximizing the first partial derivative with respect to y with respect to x. If this is done for both the $\langle 100 \rangle$ and the $\langle 11 \rangle$ defects, the following forces/atomic volume are found:

$$F^{(100)} = G\Delta\varepsilon b^4/9.45y^2 , \qquad (20)$$

$$F^{(10)} = G\Delta\varepsilon b^4 / 9.43y^2 . \qquad (21)$$

It should be noted that this quantity is essentially a constant



Figure 8. Diagram Relating the Distance of Closest Approach, y, and Distance in the Slip Direction, x, to f for a Screw Dislocation Interacting With a Defect regardless of the orientation of the defect. This means that the hardening effect of the various defects depends primarily on their size, concentration, and strength of tetragonality and not on their orientations.

If the minimum stress is rewritten in terms of the force/defect/ atomic volume, it is given by

$$\tau = Fn/b1$$
. (22)

This equation, along with the forces calculated above, and with the assumption that 1 can be found, makes it possible to calculate a value for τ .

Knowing the concentration c of defects, 1 can be calculated as

$$l = b/\sqrt{fc}$$
(23)

for interstitials, and

$$1 = b/\sqrt{fc/2}$$
(24)

for divacancies, where f is the fraction of the total concentration of defects that are interacting. By evaluating $\Delta \epsilon$ from calculations done by Huntington (14) for interstitials and from Weizer and Girifalco (15) for divacancies as 0.55 and 0.08 respectively, and by assuming y = b for distance of closest approach, the increase in stress can be calculated as

$$\Delta \tau_{\rm r} = G c^{\frac{1}{2}} / 10$$
 (25)

for interstitials, and

$$\Delta \tau_{\rm r} = G c^{\frac{1}{2}} / 100$$
 (26)

for divacancies, where

$$\Delta \tau_{\mathbf{r}} = \tau_{\mathbf{r}} - \tau_{\mathbf{o}}, \qquad (27)$$

where τ_r = resolved flow stress of the sample at any particular concentration of defects, and where τ_o = the original flow stress of the pure sample.

Fleischer's theory predicts that for equivalent concentrations of defects the interstitials will harden the sample approximately ten times more effectively than will divacancies, and that this increase in flow stress will be proportional to $c^{\frac{1}{2}}$. When the experimentally obtained flow stresses are plotted as a function of $c^{\frac{1}{2}}$ the predicted linear relationship is obtained, as shown in Figure 9. The upper line is for interstitials and the lower for divacancies. If the increase in flow stress is written as

$$\Delta \tau_{r} = (G/n)c^{\frac{1}{2}},$$
 (28)

and the equation of the $c^{\frac{1}{2}}$ lines graphed in Figure 9 as

$$\Delta \tau_r = mc^{\frac{1}{2}}, \qquad (29)$$

then

$$m = G/n$$
, (30)

where m = the slope of the $c^{\frac{1}{2}}$ line and n = the relative strength of that particular type of defect. With $G = 1.05 \times 10^3 \text{ kg/mm}^2$ the slope of the $\Delta \tau$ versus $c^{\frac{1}{2}}$ lines yield values of n = 11.8 and 43.6 for irradiated and Sr doped KCl respectively. The agreement with Fleischer's theory is satisfactory considering the approximations made.

The increase in hardness for increased concentration of tetragonal defects has been established using the resolved flow stress, τ_r , as a measure of the hardness of the sample. This τ_r was determined by using



Figure 9. The Resolved Flow Stress of Irradiated Pure KCl (upper curve) and Sr Doped KCl (lower curve) as a Function of the Square Root of the Concentration of Defects

the uniaxial compression test. Figure 10 shows the comparison of this technique with the Vickers microhardness technique. For the irradiated KCl, HV increases by 25% while τ_r increases by a factor of 8 or 9. Johnston and Westbrook (reported by Johnston and Nadeau (17)) found similar increases in HV and τ_r after irradiation of a number of alkali halides, and Chin <u>et al.</u> (18) found a significant increase in HV with increasing τ_r for the doped alkali halides. Microhardness tests activate both the primary and secondary slip systems, while uniaxial compression tests activate only the primary system; it is not surprising that the two tests yield values that are not directly proportional to each other. However, it is important to note that hardening by radiation damage and by doping yield different relations between HV and τ_r .

The wing size test makes use of motion in the primary slip system to determine the hardness of the sample. Figure 11 indicates that the dislocation rosette size, w, is directly proportional to the load on the indentor. Inspection of the figure suggests that there should be a finite wing size, w_0 , corresponding to zero load on the indentor, for each value of the flow stress. In Chapter II it was suggested that this initial wing size should be inversely proportional to the resolved flow stress. In Figure 12 a graph of τ_r versus $100/w_0$ is presented. It shows w_0 to be inversely proportional to τ_r for larger values of τ_r . w_0 seems to approach a maximum size for softer crystals. This can be expected, since for these softer crystals, w_0 is greater than 100 µm and subgrain boundaries or other macroscopic defects could easily limit its size.











Figure 12. A Plot of Resolved Flow Stress Versus 100/w o

CHAPTER V

SUMMARY

The mechanical strength of various alkali halide samples has been measured. The list of samples includes pure, irradiated, and Sr doped KC1, as well as samples of pure NaC1, pure KBr, and a $\text{KC1}_{x} \cdot \text{KBr}_{1-x}$ mixed crystal. The effect of doping with divalent ions and of irradiating the samples on the resolved flow stress has been reported and explained using Fleischer's theory. The mechanical strength of several samples was measured using four different techniques in order to compare their results. The four techniques used were the uniaxial compression test, the four point bend test, the Vickers microhardness test, and the wing size test.

The results of this comparison for the Vickers microhardness test demonstrated that while the HV number increased for an increase in flow stress, the relation between the two was not linear. When the wing size test was compared to the flow stress, an inverse relation was found for the harder samples while in the softer samples the wing size appeared to approach a maximum size. This deviation was attributed to subgrain boundaries and other macroscopic defects. A linear relation was found when the flow stress determined by the four point bend test was compared to the flow stress determined by the uniaxial compression test. Therefore, the three tests that yield a value for the flow stress of a sample are the wing size test, the four point bend test, and the uniaxial compression test. Of these three, only the wing size test is nearly nondestructive.

The resolved flow stress of pure KCl when irradiated, or when doped with the divalent ion Sr⁺⁺, was found to be proportional to the square root of the concentration of defects. It was also concluded that for equivalent concentration of defects, radiation damage hardening of the sample was more effective than was doping the sample with divalent ions. Both of these results were predicted by Fleischer's theory. However, there are several areas that should be investigated before radiation strengthened KCl is used for engineering purposes. These areas include the following: How stable are the Cl interstitials under ambient light? What is the thermal stability? How sensitive is the radiation hardening and stability to the purity of the crystal? All of these questions should be answered before use is made of radiation strengthened KCl.

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