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The Effect of Strong Electric Field on Microhardness and Mobility of the Indented Dislocations in Alkali Halides

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Some regularities and particular specialties of electromechanical effect have been investigated in NaCl single crystals. The conclusion has been drawn that the existence of the electromechanical effect in crystals with ionic interatomic bonds appears to result from both the direct electrical field's force on charged dislocations, as the filaments and the modification under field influence the impurities, and point defect's state in the crystal lattice. The last modification is to vary the interactions between the obstacles and the dislocations moving in solid, thus facilitating or hardening the microplastic deformation in the hole.

KEY WORDS Electromechanical effects, microhardness, semiconductors plastic deformation, alkali halides.

1. INTRODUCTION

The decrease in microhardness of the subsurface layers of solids exposed under the external electric fields, or electromechanical effect (EME), is observed in ionic crystals as well as in semiconductors and semimetals.^{1–13} The EME is apparently caused by the influence of the electric field on the elementary acts of crystal's plastic deformation. However, the mechanisms controlling this effect in different kinds of solids are not entirely identical. For example, in some semiconductors and semimetals the degree of manifestation of the effect seems to be closely related to the intensity of the water adsorption processes on the sample surface from the atmosphere.¹⁻³ On the contrary, in ionic crystals where EME apparently is not so sensitive to the state of the very surface (see, e.g., Reference 12), the peculiar behavior of the charged defects in the external electric field---the linear (dislocations)^{11,12} and the point (vacancies and impurities)¹³—usually plays the major role. Thus in order to understand the nature and the specific features of EME in ionic crystals, additional investigations are required. The necessity of such investigations is obvious because the main features of the EME in these crystals have not been studied in detail. There is also no complete description of the EME

micromechanisms on the atomic and molecular level taking into account the dislocation and point defect's interaction.

This paper is devoted to the further investigation of the features and nature of the EME in ionic crystals. The method of the dislocation rosettes is used here together with the measurements of the sample microhardness as in the previous work.¹²

2. EXPERIMENTAL PROCEDURE

NaCl single crystals grown by the Kyropoulos method in a vacuum were used for the investigation. Macroscopic yield point was $s_y = 250$ G/mm² (type I) and $s_y = 210$ G/mm² (type II). The samples contained both cationic (Ca⁺⁺, Mg⁺⁺, Mn⁺⁺, Fe⁺⁺) and anionic (OH⁻) impurities. The concentration of two-valent cations of each kind did not exceed $6 \cdot 10^{-4}$ at percent. The anion impurity was about $8 \cdot 10^{-3}$ at percent, thus being dominant. The initial magnitude of the dislocation's density in the crystals was not more than 10^4 cm⁻². The microhardness tester PMT-3 was used for the creation of the dislocation rosettes and for the measurements of the sample's microhardness. Indentation was made by Vickers standard diamond indenter.¹⁴ The microhardness tester was placed in a hermetically sealed air chamber. The samples were tested at room temperature and normal atmospheric pressure, and the relative humidity during experiments was kept constant (60%).

Directly before testing, the samples (plates $10 \times 10 \times 5$ mm) were prepared by chipping the primary crystal along the planes of the natural cleavage. The plates were then inserted into the chamber and exposed there for 30-40 minutes in order that the crystal's surface could reach an equilibrium state.

Still in the chamber, the samples were placed on the table of the microhardness tester between the vertical plates of a high voltage capacitor. Several series of imprints were made on the surface of each sample with and without the field action applied. In each case the plane (001) was indented. The vector of the external electric field in contrast to References 11 and 12 was always complanar to the



FIGURE 1 The dependence of imprint diagonal d on the indenter load P in the external electric field E (I) and without the field (II).

sample's surface and took its bearing along (100) direction. When all the series of imprints were made, the samples were selectively etched to reveal the developed dislocation rosettes around the imprints (see Figure 1).

The plastic deformation of the material under indentation is known to be closely related to the generation of the dislocations and to their movement in the rosette "arms." Therefore, to describe the quantitative EME—in addition—to the microhardness value H and to the imprint diagonal value d—the geometric characteristics of the dislocation rosettes, namely lengths of their edge (l_e) and screw (l_s) "arms," have been used. The distance from the rosette center up to the leading dislocation in the arm was taken as the arm length. The average of the experimental data was taken over 100 rosettes obtained under the same experimental conditions.

3. EXPERIMENTAL RESULTS

The dependence of the imprint diagonal d and the microhardness H on the indenter load P obtained for the samples indented in the external electric field of intensity E = 26 kV/cm and without the field are given in Figures 1 and 2.

It follows from Figure 1 that the dependence of the imprint diagonal on the load P is of the form:

$$P = a(E)d^{n(E)} \tag{1}$$

Without the field action, the index $n(0) = 1.83 \pm 0.02$. This result coincides with the data obtained in Reference 15 for the series of alkali-halide crystals. In the electric field E = 26 kV/cm, the parameter *n* has the value $n_E = 1.96 \pm 0.02$.

The following expression is known to be valid for the microhardness¹⁴: $H = 1854 \cdot P/d^2$, and therefore substitution of (1) in this expression gives the numerical value of H at E = 26 kV/cm that is practically independent on the indenter load (see Figure 2). The increase of the microhardness for small loads observed in alkalihalide crystals is usually related to the hardening influence of subsurface layer and



FIGURE 2 The dependence of microhardness H on the indenter load P for microindentation in the field E (I) and without it (II).

the surface of the sample.^{15–18} Therefore, invariability of the value of H under the conditions of given experiments indicates that the external electric field stimulates the processes, which partly compensate for the hardening effect of the boundary regions of the crystal.

The experiments reveal the symmetry (on average) of the edge "arms" of dislocation rosettes created in unstressed samples in the absence of the electric field influence. But the rosette's symmetry essentially falls when the strong electrical field parallel to the sample surface is applied during the indentation.

The rays of the edge "arms" with the dislocations moving in the direction of the field vector **E** turn to be longer (or shorter) on average than those directed against the field.¹⁹ Qualitative observations show that the sign and the degree of this asymmetry depend strongly on the purity of the crystal and are obviously defined by the sign and the quantity of the electric charge transferring by the dislocations. At the same time the asymmetry of the rosettes rapidly diminishes with the increasing hardness of the crystals. Additional investigations are necessary for the more detailed quantitative description of this phenomenon. Here it is worthwhile to emphasize that for NaCl single crystals used in the present work, the influence of the electric field on the edge rays directed against the field was comparatively weak. This means that in the given samples the sign of the effective charge transferring by the moving dislocations is, on average, positive due to the presence of large concentration of the anion impurities.²⁰ Therefore, the experimental data for the edge rays of these kind are excluded in the future from our consideration.

The dependencies of the lengths of the edge and the screw rosette arms on the indenter load obtained with and without the field E = 26 kV/cm are represented in Figure 3. They show that the relationship between the ray length and the load in all cases is described by the following relation



FIGURE 3 The dependence of edge (e) and screw (s) dislocation arm lengths obtained for rosettes created in the electric field (I) and without it, on the load P.

$$l = K \cdot P^m \tag{2}$$

The values of the parameters K and m for the edge (e) and screw (s) arms are determined by the method of least squares (Table I).

It is seen from the Table, that the external electric field influences only upon the K_e values, whereas the numerical values of K_s and index m are invariable in fact, the latter being slightly higher for screw arms. The numerical value for mpractically coincides with that obtained earlier for the edge rays of the rosettes in alkali-halide crystals,^{21,22} and also for the rays consisting of the 60°-dislocations in silicon.23

The experiment shows that the EME in alkali-halide crystals is observed only under microindentation with loads less then 100 G (Figure 1 and 2). In other words,

Intensity of electric field, kV/cm	K_e , 10^6 din ^{-1/2} · cm	K_s , 10 ⁶ din ^{-1/2} · cm	m _e	m_s
0	4,5 ± 0,2	2,0 ± 0,1	0,50 ± 0,01	0,53 ± 0,01
26	5,4 ± 0,2	2,1 ± 0,1	0,49 ± 0,01	0,53 ± 0,01





FIGURE 4 The dependence of relative variation in run lengths of edge leading dislocations in the electric field on the depth of semiloop's deposition (or on the load P).

plasticizing action of the electric field occurs if the surface layers of thickness not more than 100 mm are involved in the plastic deformation under indenter (see Figures 2 and 3). It is interesting to compare this result with the data on the influence of electrical field upon the run lengths of leading dislocations in the rosettes created under different indenter loads. Figure 4 shows the experimental relationship between the parameter

$$\frac{\Delta l_e}{l_e^o} = \frac{l_{e_s}^E - l_e^o}{l_e^o}$$

and the indenter load, or the same, the depth of the dislocation semiloops h.

This relationship is defined also by the power function

$$\frac{\Delta l_e}{l_e^o} = A \cdot h^{m_1} \tag{3}$$

where $m_1 = -0.4 \pm 0.01$. The experiments reveal that the value $\Delta l_e/l_e^o$ considerably depends on the crystal yield point. The less is the sample yield point the higher is numerical value of $\Delta l_e/l_e^o$. The dependence of $\Delta l_e/l_e^o$ on the intensity of the electric field is given in Figure 5 for the rosettes, created under indenter load P = 2G in relatively more soft crystals (type II). It follows from the comparison of Figures 4 and 5, that the decrease of yield point leads to the twofold increase of $\Delta l_e/l_e^o$ maximum value (at E = 26 kv/cm). In this case the dependency of $\Delta l_e/l_e^o$ on E, can be presented as

$$\frac{\Delta l_e}{l_e^o} = C \cdot E^{\alpha} \tag{4}$$

where $\alpha = 0.65 \pm 0.02$.





It should be mentioned that for the softer crystals (after annealing), the sensitivity to the electric field influence increases not only for the edge, but also for the screw dislocation arms of rosettes. However, the screw arms are as before far less sensitive to the field action, compared to the edge ones.

3. DISCUSSION

It follows from the experiments described above that the plasticizing effect of the external electric field diminishes with the increase of the indenter load, or with the depth of the subsurface layer where the dislocation semiloops are deposited. Thus the EME in ionic crystals like in semiconductors is observed only in a narrow subsurface region of the crystal. The general reasons for the EME localization in the sub-surface layer are not yet established because of the complexity of the EME mechanism itself.

Comparison of the data presented in Figures 2 and 4 shows that the lengths of the edge arms of rosettes, defined as the runs of the leading dislocations in the direction of the vector \mathbf{E} , are found to be far more sensitive to the field action than the microhardness. Therefore, it is most expedient to deal with parameters of the dislocation rosettes when EME in ionic crystals is studied. The empirical relationship, approximately describing the EME in NaCl crystals, can be obtained by the combining of the expressions 3 and 4, namely

$$\frac{\Delta l_e}{l_e} \approx \gamma \cdot 5 \sqrt{\frac{E^3}{h^2}}$$
(5)

Obviously, the parameter g is the characteristic of the material studied. For a given type of crystal the numerical value of g particularly depends on the impurity's concentration and the impurity state in the crystal lattice. More detailed information about the impurity influence on the EME will be given henceforth.

A remarkable circumstance is the nonlinear dependence of the magnitude of the effect upon the external field intensity (see expressions 5). Let's analyze this dependence in detail. The force interaction of the electric field with the dislocation, assumed to be a charged thread, is equivalent to the appearance of the additional shear stress in the planes of sliding of the dislocations²⁴:

$$\Delta \tau_{xy} \sim \rho E/b$$

where ρ is the linear charge density of the dislocation and **b** is Burgers vector.

A modelling of this effect has been fulfilled by means of making the additional mechanical stress $\Delta \tau'_{xy}$ in the slip planes of the edge dislocation arms under the one-axial compression of the sample with simultaneous inculcating of the indenter. The axis of the compression has been directed colinearly to the vector **E**. For small $\Delta \tau'_{xy}$ the following correlation takes place $\Delta I_e/l_e^o \sim \Delta \tau'_{xy}$ (see Figure 6 and Reference 25). Consequently, the value Dl_e/l_e^o would have been proportional to E only due to the direct force action of the field E on the charged dislocations. The



FIGURE 6 The relative run length variation of the edge leading dislocation in the rosette arms under the stress of one-axial compression. The axis of compression is perpendicular to the indented surface.

non-linearity of the effect observed in our experiments (see the relation 5) means that the EME in the given situation is caused not only by the above indicated force interaction. Undoubtedly, such action brings its own contribution to the EME, particularly in "pure" crystals.^{12,26}

In a roundabout way, the evidence of this conclusion is the more weak influence of the field E on the runs of screw arms—see Figure 3. Indeed, the screw dislocations under the usual conditions do not transfer the electric charge in the $\langle 100 \rangle$ direction,²⁰ and consequently they cannot be accelerated by the field E, which vector is parallel to $\langle 100 \rangle$.

However, along with the direct field-force action, the noticeable influence on the value of the effects is brought also by the field action upon the state of impurities and the other point defects that are the stoppers for the dislocations, i.e., the centers of the retardation of the dislocation motion.

It is known that under the electrical field influence, the complexes of the point defects regulate their orientation.²⁶ In addition, the point elements of these complexes are partially carried away by the elastic field of the moving dislocations,²⁷ i.e., some charged lattice defects acquire additional drift velocities. Under the favorable electric field direction both such processes lead to the decreasing of the dislocation braking by the above mentioned defects—stoppers.

Meanwhile the increasing in the dislocation velocity caused by the alteration in the defect's state appears to be a non-linear function of the external electrical field $E^{.28-31}$ Hence, the total influence of the electric field on the runs of the leading edge dislocations is found to be also non-linear.

The leading dislocations move in the rosette arm rays under the abruptly nonhomogeneous and radially decreasing stress field derived from the indenter and from the preceding arm dislocations. So the quasi-viscous mechanism of the dislocation motion that initially realizes in the rosette rays is gradually replaced by the thermally-activated mechanism of the point defects—stoppers overcoming. Accordingly, the main method of the electric field's influence upon the motion of the dislocations is also replaced. Namely, in the initial stages of rosette formation when the dislocations move in the arm rays with near-sound velocities—the basic mechanism of the electric field plasticizing is apparently the direct accelerating of the charged dislocations by the field E. In the final stages of the microindenting, when the arm dislocations move with relatively small velocities, the variations in the retarding force of stoppers under the field influence become the most important field-plasticizing mechanism.

Both these mechanisms undoubtedly define the EME value but their contributions during the various stages of imprint formation are essentially different. It is also obvious that the crystal impurities must influence the relative magnitude of these contributions.

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