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Mechanisms of the high-temperature internal stresses relaxation during creep in alkali-halide single crystals

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The process of internal stresses relaxation at jump-like change of external stresses is experimentally investigated during creep of NaCl and KCl single crystals. The basic relaxation processes have been considered and some of those have been simulated.

Экспериментально исследован процесс релаксации внутренних напряжений при скачкообразном изменении внешних напряжений во время ползучести монокристаллов NaCl и KCl. Рассмотрены основные релаксационные процессы и промоделированы некоторые из них.

Plastic deformation of single crystals is known to occur by moving of dislocations which are thermodynamically nonequilibrium defects of a crystal lattice. Therefore, a number of relaxation processes may run in dislocation subsystem even in absence of external stresses (σ) [1]. Those result in reduction of internal stresses σ_i and excess energy related thereto. The applying of external stresses activates some relaxation processes, in particular, the scattering of dislocation boundaries.

To elucidate the external stress influence on the internal stress relaxation mechanisms, experiments on jump-like change of external stresses during high-temperature deformation are of good promise. In such cases, the dislocations having an edge component of Burgers vector, may not only glide, but also climb due to diffusion. Dislocations in the external loading direction glide under action of effective stresses $\sigma_{eff} = \sigma - \sigma_i$, therefore, the analysis of kinetic creep curves shapes at partial fast unloading of samples allows to judge on time dependence of average internal stresses.

Direct dynamics observations of individual dislocations and pile-ups thereof at high temperatures is possible only in thin samples by means of an electronic microscope, while it is practically impossible in bulk crystals. Thanks to new computer technologies, it is expedient to combine experimental researches with mathematical modeling of physical processes. Comparison of these research results makes provides a better understanding of the processes occurring in dislocation subsystem of the crystals.

The experimental part of researches was carried out on alkali halide single crystals NaCl and KCl shaped as rectangular prisms of $3\times5\times10$ mm^3 with initial dislocation density $\rho_0 \approx 10^5$cm^{-2}. Crystals of two orientations <100> (crystals I) and <111> (crystals II) were strained in a temperature range of 0.6 to $0.92T_{melt}$ in the creep mode under uniaxial compression. The initial (transient) stage of creep, when changes in dislocation subsystem are the most essential, was studied. The dislocation structure of crystals prior to deformation was revealed by etch-pit method. To determine the average internal back stresses, the technique of fast changes of the applied exter-

nal stresses from σ_1 to σ_2 [2] was used (the stress interval $\sigma_1 = 0.4$ to 1.2 MPa). The values σ_i was determined from the stress σ_2 at which the deformation rate $\dot{\varepsilon}$ is equal to zero for some time, that is, σ_{eff}.

The results obtained show that σ_i increases in direct proportion to σ both for crystals I and crystals II (Fig. 1a). A higher internal stress level in crystals I (the external stresses and temperature being the same) is caused by a greater number of dislocations and their pile-ups therein as compared to crystals II, because of easier arising and sliding of dislocations in crystals I. The σ_i decreases at temperature elevation (Fig. 1b).

Experiments to determine the average internal stresses are carry out usually within several seconds or tens seconds. Study of strain curves $\varepsilon(t)$ during tens minutes after sharp stress release down to level $\sigma_2 \approx \sigma_i$ has shown interesting features: on a background of constant or decreasing strain, periodic sharp jumps of positive (in the direction of applied external loading) strains are observed, thereafter the curve $\varepsilon(t)$ gradually comes back up to the level preceding the jump (Fig. 2a). The period increases in time.

The peculiarities of kinetic creep curves after stress dumping are caused by specificity of internal stress relaxation in dislocation subsystem. At the transient creep stage, the hardening and softening processes occur at different rates [3]. The increase of internal stresses occurs due to bending of dislocation lines fixed at stoppers to radius $R \approx Gb/\sigma_1$ (G is the shear module; b, the Burgers vector value) and to formation of dislocation pile-ups at block boundaries. In the first case, the internal stresses relax after jump-like reduction of external stresses due to straightening of dislocation lines. The dislocations emitted from sources encounter during their movement under action of stresses σ the low-angle boundaries and are stopped, forming flat pile-ups. In the pile-up head (at the stopper), the stress level rises up to $\sigma_S = n\sigma$, where n is number of dislocations in the accumulation. If the stress concentration at the boundary does not exceed the stress value σ^* necessary for break of dislocations through the boundary or for its destruction, the pile-up is stopped. Between dislocations located in one slip plane, the repulsing forces act. Under equilibrium, the distance

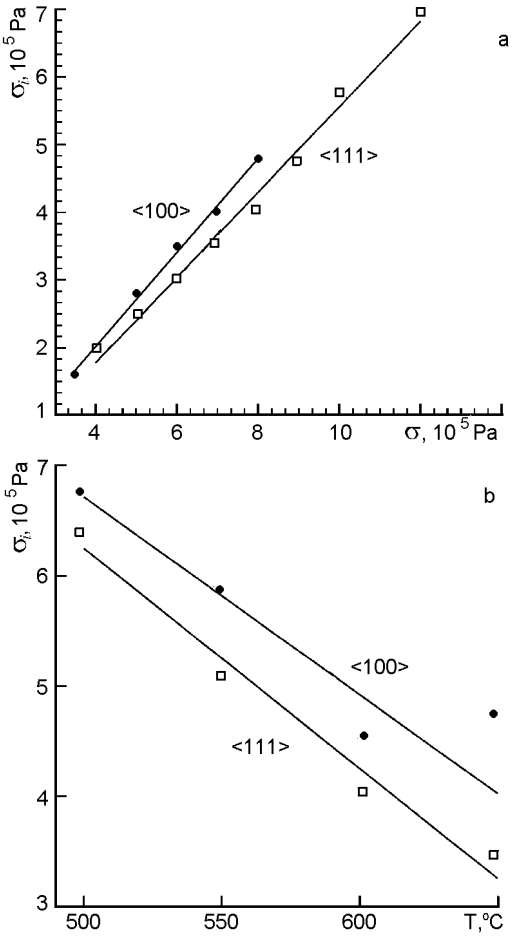

Fig. 1. (a) Dependences of internal stress value on that of external one. $T = 650$°C. (b) Temperature dependences of internal stress value. $\sigma = 0.8$ MPa. KCl single crystals.

between dislocations in the pile-up x_i are determined by [4]:

$$\sigma - \sum_{j=1}^{n} \frac{A}{x_i - x_j} = 0 \quad (i \neq j). \tag{1}$$

The pile-up equilibrium length at the certain stress is $L = 2An/\sigma$. $A = Gb/2\pi(1 - \nu)$ for the pile-up of edge dislocations and $A = Gb/2\pi$ for that of screw dislocations. As the stress level is reduced by $\Delta\sigma = \sigma_1 - \sigma_2$, the pile-up length increases by

$$\Delta L = -\frac{2An}{\sigma_1^2}\Delta\sigma. \tag{2}$$

This results in a back deformation of the crystal. From an estimation of ΔL value at known constants of a crystal, for example, KCl, at the experiment temperature of ($G = 0.85 \cdot 10^4$ MPa [5], $b = 4 \cdot 10^{-10}$ m, $\sigma^* \approx 1$ MPa),

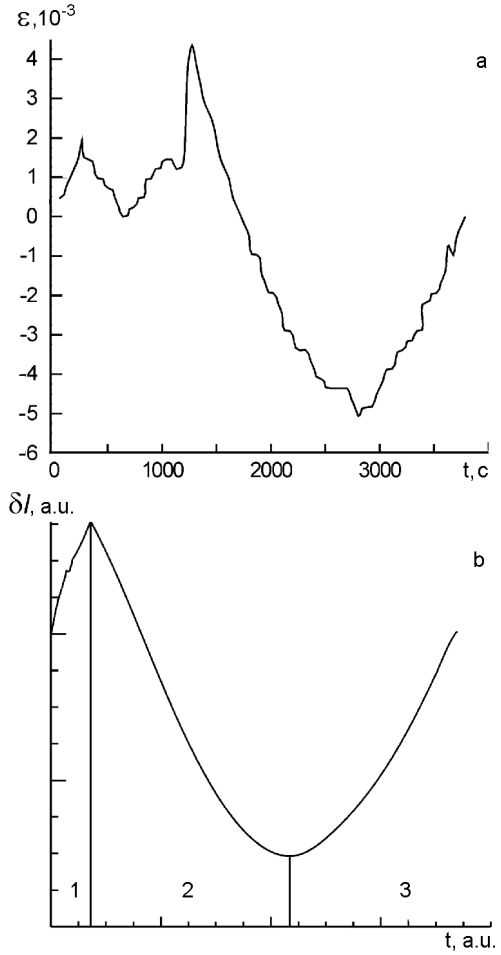

Fig. 2. (a) Experimental creep curve for KCl single crystal after external stress reduction from $\sigma_1 = 0.8$ MPa to $\sigma_2 = 0.2$ MPa. $T = 650$°C. (b) Strain curve obtained by computer simulation for break of dislocations through low-angle boundary.

it follows that at the stress dumping by 75 % (Fig. 2a), one pile-up may cause the crystal lengthening by ~2 μm, i.e. an observable back strain is caused by about 10^4 pile-ups. The number of blocks in a sample has just that order of magnitude (according to structural researches, the average block linear size l is about 100 μm). Thus, the back strain could be provided either by one pile-up in each block, or by several pile-ups in some blocks.

The non-monotonous character of kinetic creep curves (Fig. 2a) testifies to non-monotonous change of internal stresses at dumping of a part of loading, namely, to their periodic sharp reduction. As a result, the effective stresses increase, and the deformation occurs for a certain time in the direction of external stresses. There are some possible reasons for internal stress reduction. First, at high temperatures, the pile-ups of edge dislocations can extend due to diffusion climbing from the initial pile-up plane. Second, the pile-ups of screw dislocations are unstable because a cross slip is possible. However, the cross slip planes in crystals with the NaCl lattice are {100} ones, not being the easy sliding planes. Therefore, cross slip of screw dislocations is hindered and can take place if the <110> {100} sliding system becomes active. Comparison of creep rates for crystals I and II allows to conclude that at $T \geq 550$°C, dislocations in the specified slip system arise and move. The third possibility of sharp internal stress reduction is break of accumulated dislocations through dislocation boundaries. The fourth cause consists in that if the pile-up dislocations are unable to pass through the boundary, they can to destroy it and move together with dislocations of the boundary in the external loading direction. The fifth reason is that the dislocations can enter the boundary without passing through it or destroying it. In case of dislocation pile-ups stopped on both sides of boundary and having opposite Burgers vectors this process results in local reorientation of the crystal. This process is similar to formation of dump strips at plastic deformation [6]. Such areas reoriented by some degrees were observed experimentally by optical microscopy as a difference in contrast of the reflected beams and by Fujiwara X-ray technique as diffraction spot splitting [7] (Fig. 3). At last, one more possible reason of internal stress reduction consists in formation of cracks [8] in conditions when dislocation moving in easy slip systems is impossible or hindered (crystals II).

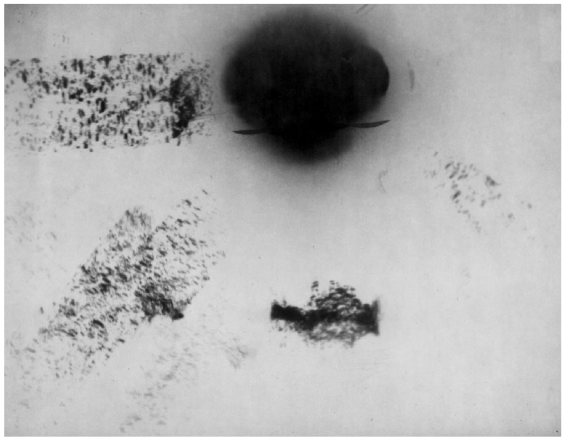

Fig. 3. Post-creep X-ray diffraction pattern of NaCl crystal obtained by Fujiwara method.

Two of the above-mentioned processes resulting in sharp internal stress reduction have been simulated. Those were a break of dislocations of an pile-up through a dislocation boundary and destruction of a dislocation boundary. In our opinion, it is just these two processes that are responsible for jump-like deformation in crystals of both orientations for the following reasons. Resolving of dislocation pile-ups due to climbing or to cross slip, and reorientation of crystal local areas result in comparatively slow internal stress reduction. Cracks are formed mainly in crystals II.

To prove that break through a dislocation boundary results in jump-like character of plastic deformation, a mathematical model of the process was constructed. We have considered not only conservative movement of dislocations, but also their ability to diffusion climbing at high temperatures. The behavior of a separate accumulation consisting of randomly distributed edge dislocations near a wall of immovable edge dislocations was simulated (the model parameters have been taken for NaCl single crystal). Such a model can be used if the curvature radius of dislocation lines exceeds considerably the average dislocation spacing. Then, the dislocations can be considered as rectilinear and the whole dislocation ensemble, when being considered, can be subdivided into subensembles, each of those being a system of parallel dislocation lines. Fig. 2b shows the creep curve obtained by computer simulation and in Fig. 4, consecutive evolution stages of a dislocation pile-up are presented. We assume that the stress dump down to σ_2 was realized at the initial moment, whereas prior to this time, the dislocation accumulations are arisen in the crystal near dislocation walls during creep (under σ_1). The creep process (curve in Fig. 2b) can be subdivided into three stages. At the first stage, deformation proceeds in the "positive" direction of the applied external stress. Dislocations which are located in the accumulation head break through the wall and move further under an external stress, till meeting another obstacle which they cannot overcome (Fig. 4, *a*–*c*). At the second stage, there is an active movement of other dislocations: "front" dislocations, towards the stopper (where those stop), "back" dislocations, in the opposite direction (where there are no obstacles). This results in a "negative" straining (Fig. 4, *d*–*e*). At the third stage, some dislocations which have left the stress field of an pile-up, move

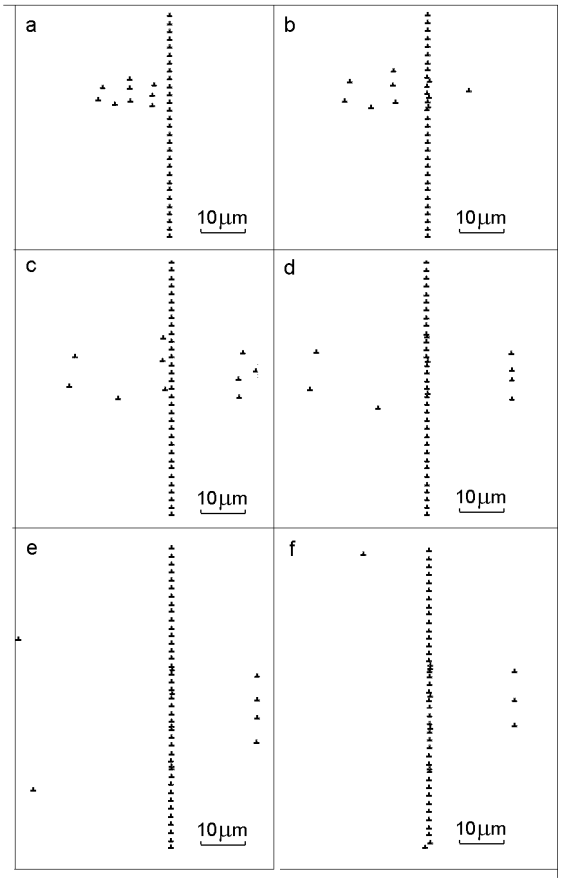

Fig. 4. Consecutive stages of evolution of dislocation pile-up near an immobile symmetric dislocation boundary.

in the "positive" direction. This process occurs simultaneously with generation of new dislocations which movement results again in "positive" deformation (Fig. 4, *e*–*f*).

Comparing the creep curves obtained experimentally and by simulation, it is seen that those are coincident qualitatively if the dominating deformation mechanism is breaking of a mobile dislocation pile-up through the array of fixed dislocation walls. Thus, it is possible to explain the non-monotony of plastic flow by the passing of previously created dislocations through fixed low-angle boundaries, thus resulting in a internal stress reduction. To provide an essential contribution of this process to creep (that could be observed experimentally), such overcoming of obstacles should either occur locally by great number of dislocations, or almost simultaneously by single dislocations in different places of the sample volume. A single dislocation provides the sample strain of $\sim 10^{-13}$ m when moving inside a block in a sample of the above-mentioned size. Thus, to provide an

effect in observable in experiment (the gauge sensitivity being ~$2{\cdot}10^{-6}$ m) at simultaneous processes in all blocks, about 10^2 dislocations should be involved in deformation (and in movement) in any of the blocks.

The second important effect influencing the non-monotonic character of plastic deformation is a scattering of dislocation boundaries when dislocations leaving the boundaries move through the block and contribute to deformation. When considering the scattering of boundaries, a special attention has been given to destruction of an asymmetric site of dislocation boundary [9]. Such sites appear always at crossing of the boundaries formed in polygonization process. The stable (that is, most energy favorable) configurations of asymmetric dislocation boundary sites have been determined. To solve that problem, the energy relief of function U(x,y) was constructed. This function is such that the force acting on unit length of the edge dislocation $\mathbf{F} = -grad\ U$. Considering the surface relief of that function, it is possible to establish the minimum and maximum values of energy barriers which generate the dislocations of different slip systems (with Burgers vectors $\mathbf{b}_1 = (b,0,0)$ and $\mathbf{b}_2 = (b,0,0)$) located within a dislocation wall so that an asymmetric site of dislocation boundary is formed. Movement of a dislocation located near to a wall is directed towards the reduction of $U(x,y)$. In Fig. 5a, the relief of function $U(x,y)$ near to the asymmetric site consisting of four dislocations with $\mathbf{b}_2$ is shown.

In the diagram presented in Fig. 5b, the arrows specify the movement directions of a dislocation with Burgers vector $\mathbf{b}_1$ near an asymmetric site containing three dislocations with $\mathbf{b}_2$. The combined consideration of such diagrams and corresponding power relieves has established that there are two stable configurations of an asymmetric site: alternation of equally spaced dislocations with $\mathbf{b}_1$ and $\mathbf{b}_2$ and a paired extremely close arrangement of such dislocations (with a possibility of dislocation reaction and formation of a sessile dislocation). Having determined possible steady configurations of asymmetric sites of dislocation boundaries, the sequence of asymmetric dislocation wall destruction has been found, that is, the dislocations for which is energy favorable to leave such boundary first have been determined.

As stated above, destruction of a dislocation wall with an asymmetric site occurs at

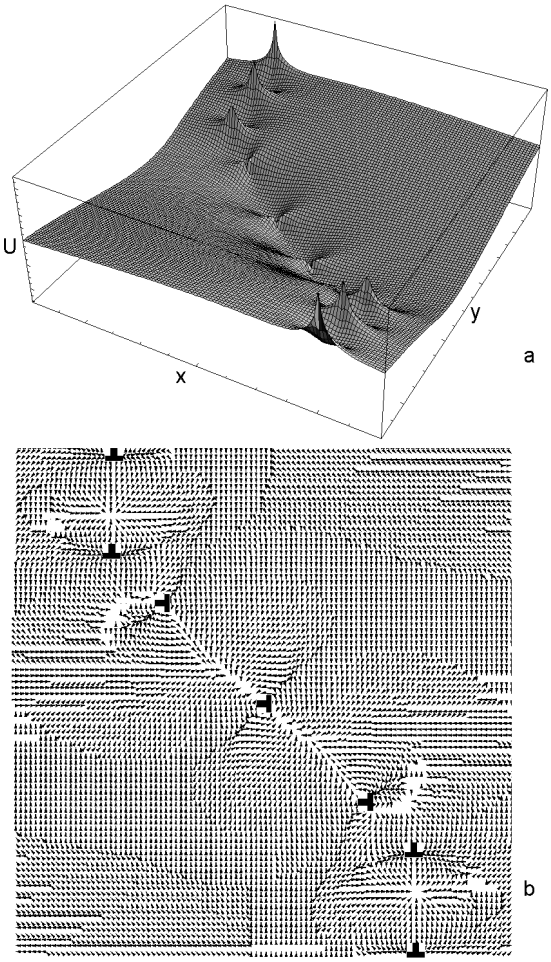

Fig. 5. (a) Energy function surface of an asymmetric dislocation wall area. (b) Diagram of possible motion directions of a dislocation near an asymmetric area of dislocation boundary.

a lower stress level than that of a symmetric dislocation wall [10]. We have calculated the value of the critical shear stress σ_{cr} necessary to destroy a dislocation wall with an asymmetric sitea, for two essentially different cases: low temperatures (dislocations move by sliding) and high temperatures ($T \sim T_{melt}$, dislocations can climb). At high temperatures, the dislocation wall with an asymmetric site is destroyed at a lower level of stresses, than at low temperatures. This can be explained by the fact that at high temperatures a dislocation can leave its slip plane and, following a minimum of a potential relief, go away from the wall.

Thus, comparison of experimental data with results of computer simulation allow to conclude that the main processes responsible for non-monotonic deformation of alkali-halide single crystals under sharp re-

duction of external stresses during high-temperature creep are processes of break of dislocations through subboundaries and destruction of dislocation boundaries.

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References

1. V.P.Matsokin, *Fuctional Materials*, **8**, 694 (2001).
2. G.N.Ahquist, W.D.Nix, *Acta Metallurgica*, **19**, 373 (1971).
3. V.M.Rozenberg, *Metalloved. i Term. Obrab.*, **7**, 89 (1973).
4. J.D.Eshelby, F.C.Frank, F.R.N.Nabarro, *Phil. Mag.*, **42**, 251 (1951).
5. A.A.Vorobyov, Mechanical and Heat Properties of Alkalihalides Single Crystals, Vysshaya Shkola, Moscow (1968).
6. J.Friedel, Dislocations, Pergamon Press, Oxford (1964).
7. T.Fujiwara, K.Koizumi, M.Hirose, Memoirs of Defense Academy Japan, **4**, 361 (1965).
8. D.V.Matsokin, I.N.Pakhomova, *Visnyk KhNU*, **651**, 136 (2005).
9. D.V.Matsokin, I.N.Pakhomova, V.P.Matsokin, *Visnyk KhNU*, **600**, 89 (2003).
10. J.C.M.Li, in: Electron Microscopy and Strength of Crystals, New York (1963).

Механізми високотемпературної релаксації внутрішніх напружень у процесі повзучості лужногалоїдних монокристалів

В.П.Мацокін, І.М.Пахомова

Експериментально досліджено процес релаксації внутрішніх напружень при стрибкоподібній зміні зовнішніх напружень під час повзучості монокристалів NaCl та KCl. Розглянуто основні релаксаційні процеси та промодельовано деякі з них.