

## THE ROLE OF CROWDION MASS TRANSFER IN RELAXATION PROCESSES NEAR HARD CONCENTRATORS

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Mechanisms of stress relaxation in KCl single crystals around hard corundum inclusions have been studied. The samples for the investigation were prepared by alloying the corundum balls into the single crystal. During the subsequent cooling of the system from the crystal melting point (1049 K) to room temperature (293 K), the ball indentation of the crystal was carried out. Dislocation structures around the balls of different sizes (from 2 to 20  $\mu\text{m}$ ) were analyzed at various cooling temperatures (cooling times were from 2 to 708 min). It has been shown that the observed number of dislocation loops (taking into account their diffusion dissolving under cooling) has transferred not more than a half of the total dilatation volume. In this connection it is assumed that not less than a half of the mass transfer process is realized by a crowdion mechanism.

### INTRODUCTION

In view of current requirements to strength, reliability and durability of materials, appropriate investigations are actual and important. The strength is determined not only by interatomic bond forces in the material.

This depends on existence of structure inhomogeneities (inclusions, pores, cracks, etc.) both occasional and specially made; these, on the one hand, result in concentration of external loading stress starting the relaxation processes of mass transfer by diffusion, dislocation or crowdion mechanisms [1–3] which admit formation of secondary cracks lowering the material strength. On the other hand, hard inclusions hamper the movement of dislocations as plasticity carriers thus strengthening the material [4].

The second principal thing determining the strength is caused by statistical processes [5, 6] connected with atomic thermal movement and energy fluctuations which result in formation and accumulation of structure changes lowering the strength under subthreshold loads. The destruction under subthreshold loads is a kind of a kinetic process coming on after some time called durability. The passing of this latent process is in fact the evolution of a synergetic system; during its self-organization the critical state and subsequent destruction take place [7]. As temperature and load increase, everything else being equal, the durability drops.

Deformation processes and mass transfer near local inhomogeneities are relevant to technologies of materials with high properties (powder metallurgy [8], diffusion welding [9], nano-technologies [10], precipitation hardening [11–13], etc.). These processes are the basis of such technologies, so are the subject of constant attention.

However, much remains unknown, in particular, as to the role of the crowdion mechanism. For example, in [14–16] it was shown for the first time that under optical breakdown of alkali halid single crystals by the laser impulse, at the process beginning, 95% of the substance from a formed void is taken away by crowdions at the front of a supersonic blast wave; that is possible only for crowdions [17, 18]. The crowdion mass transfer takes

place also in other cases [19, 20] not connected with high impulsiveness.

In the present work we carried out experiments where the rate of relaxation process can be varied in a rather wide range in order to reveal possible changing the contribution ratio of different mass transfer mechanisms under variation of the process “impulsiveness”.

### METHOD AND EXPERIMENT

The idea of the experiment follows experiments on optical breakdown of KCl single crystals by laser light [14–16]. For the present investigation we also have chosen KCl single crystals which are applied widely in IR-optics, laser technique, and instrumentation. Being sufficiently strong, low hygroscopic, easy treated and etched for dislocations, etc., the KCl single crystals are convenient for experiments. The results on them are always correct, reliable, so, are applicable for generalizing. Therefore KCl single crystals as well as copper are often used as model objects in experiments.

A corundum ball with low linear expansion coefficient  $\alpha_{sf} = 8,4 \cdot 10^{-6} \text{ deg}^{-1}$  and high elasticity modulus  $E_{sf} = 55 \cdot 10^{10} \text{ N/m}^2$  [21], in our case, plays a role of a plasmoid under high pressure deforming the crystal. The KCl single crystal has substantially larger linear expansion coefficient ( $\alpha_{cr} = 37,4 \cdot 10^{-6} \text{ deg}^{-1}$  at 50 °C and  $\alpha_{cr} = 59,7 \cdot 10^{-6} \text{ deg}^{-1}$  at 735 °C) and lower elastic modulus  $E_{cr} = 29,67 \cdot 10^9 \text{ N/m}^2$ . The ball was inserted into the KCl single crystal at pre-melting temperature. During cooling the system from KCl melting point ( $T_m = 1049 \text{ K}$ ) to room temperature  $T_{room} = 296 \text{ K}$ , the defect of the void volume occurred, and the corundum ball deformed the soft crystal by expanding the void and lowered the stress in the system. The rate of the cooling can be varied in a fairly wide range, thus varying the level of the process “impulsiveness”.

The shapes of the cooling curves constructed by points according to the equation:

$$T = T_m e^{-\alpha t}, \quad (1)$$

are shown in Fig. 1. The cooling constant  $\alpha$  (and corresponding time of cooling to room temperature) has a set of values:  $\alpha_1 = 3.0 \cdot 10^{-5} \text{ c}^{-1}$  (708 min),  $\alpha_2 = 1.05 \cdot 10^{-4} \text{ c}^{-1}$  (202 min),  $\alpha_3 = 1.05 \cdot 10^{-3} \text{ c}^{-1}$  (20 min),  $\alpha_4 = 1 \cdot 10^{-2} \text{ c}^{-1}$  (2 min).

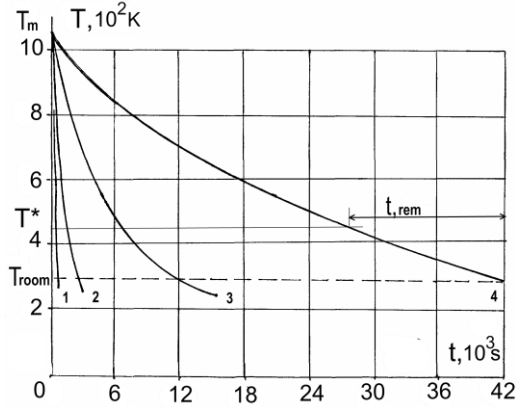


Fig. 1. Crystal cooling curves by exponential law  $T = T_m \exp(-\alpha t)$  in the range from  $T_m$  to  $T_{room}$  with cooling constants  $\alpha$ : 1 –  $1 \cdot 10^{-2} \text{ s}^{-1}$ ; 2 –  $1.05 \cdot 10^{-3} \text{ s}^{-1}$ ; 3 –  $1.05 \cdot 10^{-4} \text{ s}^{-1}$ ; 4 –  $3 \cdot 10^{-5} \text{ s}^{-1}$

In the initial state, at  $T_m$ , the volumes of the ball  $V_{cr}$  and the void  $V_{sf}$  are equal and taken as initial  $V_{sf} = V_{cr} = V_0$ . Firstly, in order to determine the stress after cooling by  $\Delta T$  (in the absence of relaxation), we consider a free ball out of the void, and a free single crystal with a void (without a ball). Relative volume changes of the void and the ball after cooling by  $\Delta T$  are the following:

$$\begin{aligned} (\Delta V_{cr} / V_0) &= 3\alpha_{cr} \Delta T \text{ and} \\ (\Delta V_{sf} / V_0) &= 3\alpha_{sf} \Delta T. \end{aligned} \quad (2)$$

The total discrepancy of the void and ball volumes  $\Delta V_d$  after cooling by  $\Delta T$  gives

$$(\Delta V_d / V_0) = 3\Delta T(\alpha_{cr} - \alpha_{sf}). \quad (3)$$

In real, the ball being in the void interacts with the crystal; therefore, the total discrepancy  $(\Delta V_d / V_0)$  should be redistributed between the ball  $(\Delta V_{dsf})$  and the void  $(\Delta V_{dcr})$ . In order to determine these values let us simulate the following: apply to the free ball the external pressure  $P'_d$  need [22] to decrease the ball volume by the total discrepancy  $(\Delta V_d / V_0)$  corresponding to the temperature decrease by  $\Delta T = T_m - T_{room}$ :

$$P'_d = K_{sf} \frac{\Delta V_d}{V_0}. \quad (4)$$

Then put the ball into the void and take off the pressure. Restoring its volume the ball presses onto the void surface, therefore the total discrepancy  $(\Delta V_d / V_0)$  due to the partial deformation of the void in the crystal and the ball would be redistributed between the crystal and the ball for achieving equilibrium when the pressure onto the void surface  $P_{dcr}$  and onto the ball surface  $P_{dsf}$  become equal, i. e.:

$$P_{dcr} = P_{dsf} = P_d. \quad (5)$$

Numerical value  $P_d$ , evidently, is much lower than  $P'_d$ .

Taking into account (4) and (5) we can write:

$$K_{cr}(\Delta V_{dcr} / V_0) = K_{sf}(\Delta V_{dsf} / V_0), \quad (6)$$

where  $\Delta V_{dcr}$  and  $\Delta V_{dsf}$  are volume changes of the void and the ball respectively in the state of elastic equilibrium of the 'crystal-ball' system. At that, the sum of the volume relative variations for the void  $(\Delta V_{dcr} / V_0)$  and for the ball  $(\Delta V_{dsf} / V_0)$  should be equal to the total discrepancy  $(\Delta V_d / V_0)$  under their independent and similar cooling by  $\Delta T$ :

$$(\Delta V_{dcr} / V_0) + (\Delta V_{dsf} / V_0) = (\Delta V_d / V_0). \quad (7)$$

From (6) and (7) we obtain the parameters of the stress state in the crystal after cooling the 'crystal-ball' system by  $\Delta T$  in the absence of relaxation processes:

$$\frac{\Delta V_{dcr}}{V_0} = \frac{K_{sf}}{K_{cr} + K_{sf}} \cdot 3\Delta T(\alpha_{cr} - \alpha_{sf}), \quad (8)$$

$$P_d = \frac{K_{cr} \cdot K_{sf}}{K_{cr} + K_{sf}} \cdot 3\Delta T(\alpha_{cr} - \alpha_{sf}). \quad (9)$$

The finite values of  $(\Delta V_{dcr} / V_0)$  and  $P_d$  after cooling the system from  $T_m$  to  $T_{room}$  are the following:  $(\Delta V_{dcr} / V_0)|_{room} = 0.086$ , while the pressure in the absence of relaxation processes would be  $P_d|_{room} = 2.15 \cdot 10^9 \text{ N/m}^2 = 2.15 \cdot 10^4 \text{ atm}$ , i. e. under lowering the temperature, the rate of the pressure increasing is  $dP / dT \approx 28.5 \text{ atm/K}$ .

The field of mechanical stress in the crystal with a void being under hydrostatic pressure  $P$ , as it follows from calculations [22, 23], is described by the tensor:

$$\sigma_{ik} = -P \frac{R_0^3}{2r^3} \left( \delta_{ik} - \frac{3x_i x_k}{r^2} \right). \quad (10)$$

The pressure  $P$  is determined by the equation (9). The Eq. (10) describes the pressure in the crystal around the dilating inclusion (the ball in our case). The components of shear stress for  $\sigma_{xy} = \sigma_{yx}$ , for example, can be written as:

$$\sigma_{xy} = \frac{3}{2} R_0^3 \frac{xy}{(x^2 + y^2)^{5/2}} P. \quad (11)$$

The stresses reach the maximum  $\sigma_{xy} = \frac{3}{4} P$  at the contour with radius  $(R_0 / \sqrt{2})$  on the sphere surface (Fig. 2,a) and drop quickly with the distance  $y$  from the void by the law  $\sigma_{xy} \sim P(R_0 / y)^4$ .

In Fig. 2,a simplistic scheme is shown for generation and evolution of prismatic dislocation loops on the void surface.

As the stresses in the crystal at the boundary with the ball on the circle contour with radius  $R_0 / \sqrt{2}$  reach the threshold values for dislocation generation, a shear loop is generated (see Fig. 2,a), its screw parts bypass the slip cylinder and move towards each other, get together and annihilate forming two prismatic loops: a vacancy one remaining on the void surface and increas-

ing its volume, and an interstitial loop stretching into the crystal depth.

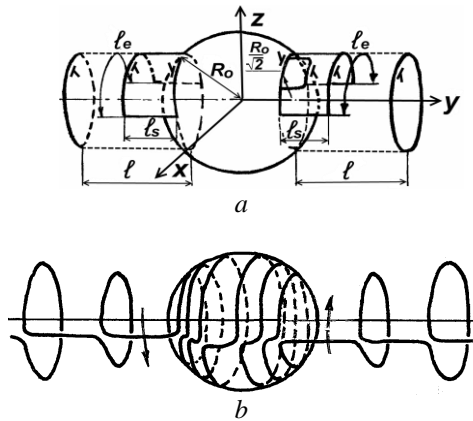


Fig. 2. Schematic representation for generation and evolution of prismatic dislocation loops:  
*a* – homogeneous generation;  
*b* – heterogenic generation

The process shown in Fig. 2,a, and called homogeneous generation of a dislocation loop, as calculations point, is possible under high stress about the theoretical strength of the crystal [24]. Another process possible under stress near threshold value for dislocation movement is shown in Fig. 2,b. In this case, necessary prismatic loops occur from a part of a growth screw dislocation moving in the field of shear stress created by an inclusion over the slip cylinder (see Fig. 2,b); formed helices have turns equivalent to prismatic loops of the types mentioned.

As dislocations are accumulated in the cluster, reverse stresses increase around the void in the direction from the dislocation cluster to the source, that's why for generation of new dislocations it is necessary to overcome not only threshold stresses, but also the reverse stresses of the cluster.

## RESULTS AND DISCUSSION

The examples of most typical structures formed near the balls at different cooling rates are shown in Fig. 3. Near the balls of small sizes about 2 to 5  $\mu\text{m}$ , dislocation cruciform rosettes are observed. At low cooling rates, pair matching of etch pits is seen. In the surrounding area of larger balls, the crosswise of the rosettes manifests clearly at low cooling rates. At high rates, the crosswise occurs at the background of increased dislocation density in the circular area around the ball (see Fig. 3).

Quantitative treatment of dislocation structures (similar to that shown in Fig. 3) occurred around the balls of different sizes and at different cooling rates was carried out to determine the relative volume proportion ( $\Delta V_{\perp} / \Delta V_d$ ) of dislocation loops remained after cooling ( $\Delta V_{\perp}$ ) to the value of the total volume discrepancy ( $\Delta V_d$ ) of void and ball sizes at room temperature. The results of the treatment are given in Fig. 4.

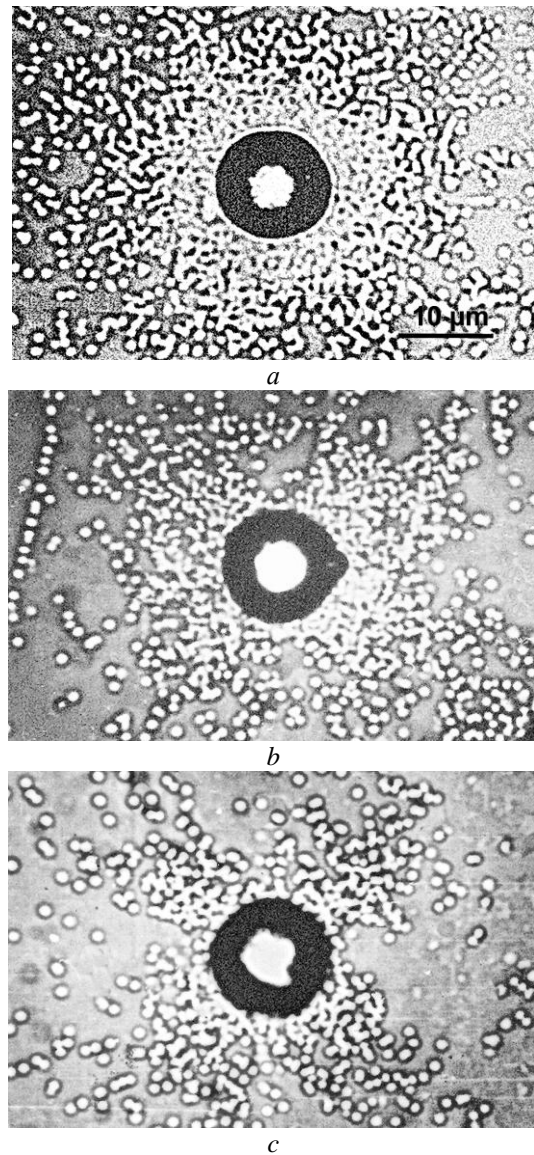


Fig. 3. Dislocation structures in the (100) plane around corundum balls in KCl single crystals after cooling by the exponential law from  $T_m$  to  $T_{room}$  with cooling constant  $\alpha$ :

$$a - 1 \cdot 10^{-2} \text{ s}^{-1}; b - 1.05 \cdot 10^{-3} \text{ s}^{-1}; c - 3 \cdot 10^{-5} \text{ s}^{-1}$$

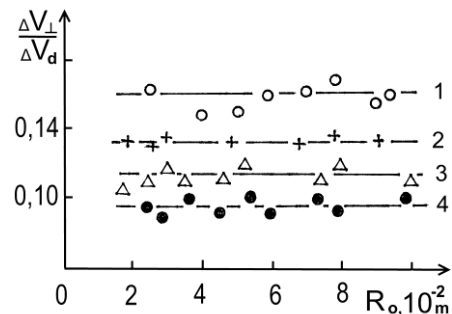


Fig. 4.  $\Delta V_{\perp} / \Delta V_d$  ratios versus radius  $R_0$  of the corundum ball after cooling the crystal from  $T_m$  to  $T_{room}$  by the exponential law with cooling constant  $\alpha$ :  
 1 –  $1 \cdot 10^{-2} \text{ s}^{-1}$ ; 2 –  $1.05 \cdot 10^{-3} \text{ s}^{-1}$ ; 3 –  $1.05 \cdot 10^{-4} \text{ s}^{-1}$ ;  
 4 –  $3 \cdot 10^{-5} \text{ s}^{-1}$

For analysis of the dislocation structure formation and evolution, it is necessary, clearly, to take into consideration the “impulsiveness” of the process, attained by variation of loading rate in the fairly wide range. The situation like this was studied in [25] with impulse loading under conditions of NaCl single crystalline samples expansion. It was shown that independently on the loading increase rate, dislocations move by jumps between the points of fixing by point defects (pinning). The pinning stress  $E_{pin}$  was found to be not more than  $0.1 \sigma_p$  (yield strength), so, did not affect decisively on the geometry of the dislocation cluster.

In our case, a locally inhomogeneous system is considered in the regime of temperature lowering when the stress around a foreign inclusion (ball) increases and, independently on the character of dislocations movement and absence of sinks for them, the cluster forms with its reverse stress restraining the emission of new dislocations. Further growth of stress caused by continued temperature decrease, diffusion dissolving the dislocation loops, and thermo-fluctuation spread of the cluster promote its softening which results in generation of new dislocation loops and further stress relaxation around the inclusion under cooling. Such the process continues to the full cooling of the system.

As it follows from Fig. 4, only from 10 to 15% of volume from the total discrepancy ( $\Delta V_d$ ) are compiled in the dislocation structures observed (independently on the particle sizes). At high cooling rates the ratio  $\Delta V_{\perp} / \Delta V_d$  is somewhat larger.

Qualitatively, the reason of such results may be caused by the fact that dislocation loops being at high temperature are diffusion dissolved and escape during cooling.

The lower are cooling rates, the more time of staying at high temperature; hence more loops have time to be dissolved than at high cooling rates (see Fig. 4). However, it would seem to be expected that the smaller are the balls (i. e. the loops are smaller), the quicker loops dissolving, and so, less quantity of loops remains. But that is not observed.

Another reason of the lowered quantity of dislocation may be connected with existence also another mechanism of mass transfer from the stressed area near the ball into the crystal depth.

Let us discuss the dislocation mechanism of mass transfer taking into account diffusion dissolving the interstitial dislocation loops. The rate of the dislocation loop dissolution is described by the relation [26]:

$$\frac{dR_l}{dt} = - \frac{2\pi}{b \ln 8R_l/b} \times \left\{ \frac{Gb [\ln R_l/b + g] D\omega}{4\pi (1-\nu) R_l kT} + D_V \Delta C_V \right\}, \quad (12)$$

where  $R_l$  is the dislocation loop radius;  $b$  is Burgers vector;  $G$  is shear modulus;  $g \approx 2...3$  is the value accounting the energy of a dislocation nucleus;  $D$ ,  $D_V$  are diffusion coefficients for atoms and vacancies, respectively (at the temperature  $T$ );  $\omega$  is atomic volume;  $\nu$  is Poisson coefficient;  $k = 1.38 \cdot 10^{-23}$  J/K

is Boltzmann constant;  $C_V$  is vacancy equilibrium concentration far from the loop;  $\Delta C_V$  is vacancy supersaturation value.

Under real conditions, taking  $\ln 8R_l/b \approx \ln R_l/b + g$ , we obtain:

$$\frac{dR_l}{dt} \approx - \frac{GD\omega}{2(1-\nu)R_l kT} - \frac{2\pi D_V \Delta C_V}{b \ln 8R_l/b}. \quad (13)$$

The first summand in equations (12) and (13) is the dissolution rate caused by loops curvature, the second is caused by supersaturation of the lattice by vacancies. We discuss each of the summands. The first summand is easy to be written as the rate of the loop volume decreasing ( $dV_l/dt$ ). Taking the loop volume  $V_l$  equal to  $\pi R_l^2 b / 2$ , we obtain:

$$\frac{dV_l}{dt} = - \frac{GD\omega\pi b}{1-\nu kT}. \quad (14)$$

In writing  $V_l$  we have taken into account that, as it was mentioned above, loops are generated over the contour of maximum shear stress with radius  $R = R_0 / \sqrt{2}$ ,  $R_0$  is the ball (void) radius.

The problem on formation and evolution of a dislocation assemble in relaxation processes near local inhomogeneities under non-stationary conditions of the system cooling is quite cumbersome and complex, especially taking into consideration that it is described by time varying external conditions and dynamical parameters.

The preliminary qualitative analysis and available information [15, 16, 19] on relaxation processes under similar conditions show that the dislocation-diffusion mechanism, as a rule, is not the only realized mechanism of mass transfer.

In this connection, we have considered reasonable to make numerical estimations for key parameters of the system during its cooling; on this basis we can quite reliably estimate quantitatively (taking into account the loops diffusion dissolution and data from Fig. 3) the total contribution of the diffusion-dislocation mechanism under conditions described; that would give a possibility to estimate quantitatively the contributions of other mechanisms as well.

It is possible to say confidently, that at high temperature when diffusion mobility of atoms is high, dislocation loops, being generated, are dissolved quickly (Table). However, beginning with some temperature, the loops (with sizes studied here) can not dissolve entirely during the remaining cooling time. We have chosen several temperature points on the cooling curves:  $T^* = 823; 773; 723; 673; 623; 573; 527; 479$  K and calculated corresponding diffusion coefficients  $D(T^*)$  using the numerical value  $D$  for 823 K experimentally obtained in [27] for similar single crystals KCl:  $D_{823} = 2.68 \cdot 10^{-14}$  m<sup>2</sup>/s; the activation energy 2.2 eV has been taken from [28]. Then we've calculated the rates of loop dissolving  $dV_l/dt$ , dissolution time  $t_{dis}$  for loops with average radius  $R_l = 3 \cdot 10^{-6}$  m, and time  $t_{rem}$  remaining for cooling from each temperature  $T^*$  to room  $T_{room}$  for all values of the cooling parameter  $\alpha$ , s<sup>-1</sup> (see Fig. 1). For convenience of the analysis and visibility, all numerical values are given in Table.

Temperature , $T^*, K$	Diffusion coefficient, $D, m^2/s$	Cooling time (s) from $T^*$ to $T_{room}$ at given $\alpha$ ( $s^{-1}$ ) parameter				Time of the loop dissolution $t_{dis}, s$
		$\alpha=3 \cdot 10^{-5}$	$\alpha=1.05 \cdot 10^{-2}$	$\alpha=1.05 \cdot 10^{-3}$	$\alpha=1 \cdot 10^{-2}$	
1049 ( $T_m$ )	–	$42.51 \cdot 10^3$	$12.15 \cdot 10^3$	$1.215 \cdot 10^3$	127	–
823	$2.68 \cdot 10^{-14}$	$34.40 \cdot 10^3$	$9.84 \cdot 10^3$	$0.98 \cdot 10^3$	103	8
773	$3.90 \cdot 10^{-15}$	$32.34 \cdot 10^3$	$9.24 \cdot 10^3$	$0.92 \cdot 10^3$	97	50
723	$3.69 \cdot 10^{-16}$	$30.10 \cdot 10^3$	$8.60 \cdot 10^3$	$0.86 \cdot 10^3$	90	500
673	$2.70 \cdot 10^{-17}$	$27.72 \cdot 10^3$	$7.92 \cdot 10^3$	$0.79 \cdot 10^3$	83	6350
623	$1.28 \cdot 10^{-18}$	$25.15 \cdot 10^3$	$7.18 \cdot 10^3$	$0.72 \cdot 10^3$	75	$1.24 \cdot 10^5$
573	$3.60 \cdot 10^{-20}$	$22.36 \cdot 10^3$	$6.39 \cdot 10^3$	$0.64 \cdot 10^3$	67	$4.06 \cdot 10^6$
523	$4.80 \cdot 10^{-22}$	$19.30 \cdot 10^3$	$5.50 \cdot 10^3$	$0.55 \cdot 10^3$	58	$2.80 \cdot 10^8$
473	$2.89 \cdot 10^{-24}$	$15.96 \cdot 10^3$	$4.55 \cdot 10^3$	$0.46 \cdot 10^3$	48	$4.17 \cdot 10^{10}$

From data of Table it follows that dissolution time for dislocation loops down to the temperature near 773 K is substantially less than the time remaining for the system cooling at any chosen parameters  $\alpha$ ; consequently, all the generated before  $T = 773$  K loops should be entirely dissolved. As the temperature decreases, the dissolution time increases exponentially, so, near 623 K all the generated loops remain practically undissolved (see Table).

At the temperature 723 K, according to the Table it is expected entire dissolution of loops under cooling with  $\alpha = 3 \cdot 10^{-5} s^{-1}$  and with  $\alpha = 1.05 \cdot 10^{-4} s^{-1}$ . At 673 K, the loops have time to dissolve only under the slowest cooling with  $\alpha = 3 \cdot 10^{-5} s^{-1}$ . In the Table the areas of dissolution and indissolubility are separated by a bold stair-step line. As it is seen from Table, least of all of undissolved loops remain under the slowest cooling; that is confirmed also by Fig. 4.

As well, quantitative estimations can be done for the portions of dissolved and remained loops. Indeed, cooling temperature entire variation corresponding to formation of total discrepancy of the void and the ball radii,  $\Delta V_d$ , is  $\Delta T = 1049 - 293 = 756$  K. The pressure within the void occupied by the ball increases during cooling with the rate  $\Delta P/\Delta T = 28.5$  atm/K (see Eq. (9)). If we neglect both the threshold pressure for dislocation generation and the threshold pressure when approaching the room temperature (and dislocation generation stops), it could be considered that dislocation loops generation and total dissolution take place in the temperature range from 1049 to 773 K, i. e.  $\Delta T = 276$  K according to the Table. This corresponds to the fraction of eliminated discrepancy about  $\Delta V_{d,dis}/V_0 = 276/(1049 - 293) = 0.365$ . As it was noticed above, the dislocations generated at  $T = 623$  K and lower remain undissolved under cooling to  $T_{room} = 293$  K. This corresponds to the fraction of discrepancy  $\Delta V_{d,rem}/V_0 = (623 - 293)/756 = 0.437 \dots$

Between temperatures 773 and 623 K, dislocations are generated and partially dissolved, but it is difficult to determine how many of them are dissolved and how many remained. This temperature range corresponds to the fraction of discrepancy  $\Delta V_d'/V_0 = 773 - 623 / T_m - T_{room} \approx 0.2$ .

Additionally, the following is be taken into account. In our estimations we neglect the role of the second summand in Eq. (12) and (13) which considers the in-

fluence of possible vacancy supersaturation on dissolution of interstitial loops. In this connection we notice the following. During local plastic deformation, generated shear loops (see Fig. 2) are intersect each other thus generating point defects – vacancies and interstitial atoms. At that, the number of interstitials is found to be more [24]; therefore, some supersaturation of interstitials can occur which would even hamper a little the dissolution of interstitial dislocation loops. When discussing the estimations above, we can neglect this and consider that not less than 50% of dislocation would remain from the quantity need for stress relaxation, if excess substance is removed from the void only by interstitial dislocation loops.

Really, according to Fig. 4, only from 10 to 15% dislocations remain. With considering given above and data of Fig. 4, we can only say, that in the stress relaxation process, besides the dislocation-diffusion mechanism, also other mechanisms of mass transfer take part. These other mechanisms may be both interstitial and crowdion ones. For ionic crystals NaCl, the vacancy diffusion mechanism is specific. An argument for this is sharp increase of diffusion coefficient in the low-temperature range in the presence of a divalent cation impurity in the crystal (for example, Ca, Mg, Ba, etc.). In the crystals used here, their concentration is not more  $\approx 10^{-5}$  mol.%. The increase of diffusion coefficient  $D$  was observed only in the temperature range lower than  $\approx 673$  K [28]. In this range  $D$  is so small (see Table) that the diffusion contribution can be neglected at all.

On the basis of above, we should consider that it is the crowdion mass transfer that would able to provide the observed quick kinetics of relaxation processes especially in the beginning stage of cooling when the cooling rate  $dT/dt$  is maximal. This result agrees with numerous observations known from literature: under micro-indentation [19, 29], under formation of a contact area during the diffusion welding [30], in healing the cracks [31]. In [32], for example, the crowdion participation in the nano-indentation process was strongly enough motivated by the fact that the activation volume determined during evolution of the indenter imprint was  $10^{-30} m^3$  (about atomic volume) which testified in favor of the crowdion mechanism. During further indentation, the activation volume increased to  $10^{-28} m^3$  that was typically for the dislocation mass transfer. The crowdion

mass transfer was observed in the very beginning stage also in [16].

If assuming in our case that the dislocation mechanism is also activated at the end of the process, the observed quantity of remained dislocations is the total contribution of the dislocation mechanism.

## CONCLUSIONS

A series of experiments has been carried out on studying the mechanisms and kinetics of stress relaxation in KCl single crystals around the fused into the single crystal corundum balls under cooling the system from KCl melting point (1049 K) to room temperature (293 K). From obtained images of dislocation structures it follows that the present quantity of dislocations corresponds only to 10...15% of total transferred volume during relaxation under cooling. Considering the quantity of diffusion dissolved dislocation loops we obtain additionally  $\approx 35\%$ . Thus, not less 50% of the mass transfer is accounted for the assumed crowdion mechanism.

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## **РОЛЬ КРАУДИОННОГО МАССОПЕРЕНОСА В РЕЛАКСАЦИОННЫХ ПРОЦЕССАХ ВБЛИЗИ ЖЕСТКИХ КОНЦЕНТРАТОРОВ**

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Исследованы механизмы релаксации напряжений в монокристаллах KCl вокруг жестких сферических включений из корунда. Образцы для исследования получались методом сплавления шариков корунда в монокристалл. В процессе последующего охлаждения системы от температуры плавления кристалла (1049 К) до комнатной (293 К) происходило индентирование кристалла шариком. Анализируются дислокационные структуры вокруг шариков разных размеров (от 2 до 20 мкм) при различных скоростях охлаждения (время охлаждения от 2 до 708 мин). Показано, что наблюдаемым количеством дислокационных петель (с учетом их диффузионного растворения в процессе охлаждения) перенесено не более половины всего объема дилатации. В связи с этим предполагается, что не менее половины требуемого массопереноса осуществлено краудионным механизмом.

## **РОЛЬ КРАУДИОННОГО МАССОПЕРЕНОСУ В РЕЛАКСАЦІЙНИХ ПРОЦЕСАХ ПОБЛИЗУ ЖОРСТКИХ КОНЦЕНТРАТОРІВ**

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Досліджені механізми релаксації напружень у монокристалах KCl навколо жорстких сферичних включень з корунду. Зразки для дослідження отримувались методом сплавлення кульок корунду в монокристал. У процесі подальшого охолодження системи від температури плавлення кристала (1049 К) до кімнатної (293 К) відбувалося індентування кристала кулькою. Аналізувалися дислокаційні структури навколо кульок різних розмірів (від 2 до 20 мкм) при різних швидкостях охолодження (час охолодження від 2 до 708 хв). Показано, що спостережуваною кількістю дислокаційних петель (з урахуванням їхнього дифузійного розчинення в процесі охолодження) перенесено не більше половини всього об'єму дилатації. У зв'язку з цим припускається, що не менше половини необхідного масопереносу здійснено краудионним механізмом.