Functional Materials 20, No.1 (2013)

Kinetics for dislocation structure formation in contact area of squeezed crystalline solids

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Received December 18, 2012

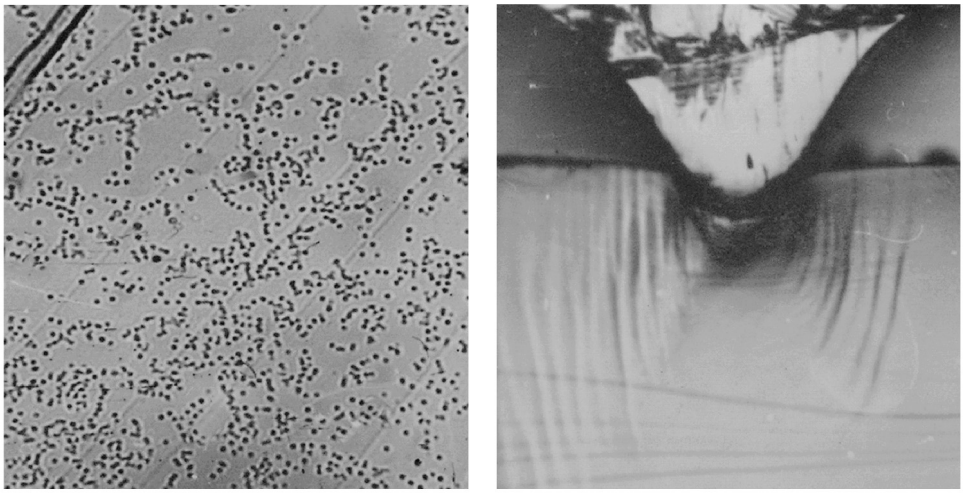
Evolution of dislocation structure during formation of the contact area was studied in modeling experiments "wedge-plane" for KCl single crystal under loading at room and high ($0.8T_m$) temperatures. It was shown that at the first (rapid) stage of the process, a quasi-stationary dislocation assemblage is formed due to emission of dislocations. It was established that at high temperature ($0.8T_m$), the cellar structure (CS) was formed at the earliest stages of annealing under loading. The experimentally determined dislocation critical density ρ_c at which the CS is formed $\rho_c \approx 10^7$ cm^{-2} is by the order of value corresponds to the calculated one ($10^6 < \rho_c < 10^{10}$) cm^{-2}. The crystal-likeness of the CS predicted by the theory is supported qualitatively; the CS contains a mixture of cells-polygons with the number of sides predominantly from 4 to 6.

Экспериментально исследована эволюция дислокационной структуры при формировании контакта в модельных экспериментах "клин-плоскость" из монокристалла KCl под нагрузкой при комнатной и высокой ($0.8T_m$) температурах. Показано, что на первой (быстрой) стадии процесса за счет эмиссии дислокаций формируется квазистационарное дислокационное скопление. Установлено, что при высокой температуре ($0.8T_m$), ячеистая структура (ЯС) формируется на самых ранних этапах отжига под нагрузкой. Экспериментально определена критическая плотность дислокаций ρ_c, при которой ЯС формируется: $\rho_c \approx 10^7$ см$^{-2}$, что по порядку величины соответствует ее расчетному значению ($10^6 < \rho_c < 10^{10}$) см$^{-2}$. Кристаллоподобие ЯС, предсказываемое теорией, качественно подтверждается; ЯС содержит смесь ячеек — многоугольников с числом сторон преимущественно от 4 до 6.

1. Introduction

Interaction of the solids being in contact and formation of the contact area between the solids under squeezing is a multidimensional problem of both great scientific and practical importance because that is a basis of various technologies for production of new materials and items. Contact surfaces of solids keep some roughness even after the most thorough treatment, thus under squeezing, a lot of mechanical stress local concentration sites occur; their relaxation results in contortion and embedding of the roughness followed by mass-transfer process by different physical mechanisms and formation of the contact area between the solids.

The mass-transfer mechanisms have been studied for a number of decades and are of interest nowadays due to great importance for modern technologies. Among the achievements of the last decade, the following developments can be mentioned: the conception on dislocation structure (DS) evolution under deformation [1–5]; mechanisms and kinetics of mass-transfer in local stressed areas occurring near dispersion in-



a b

Fig. 1. (a) Dislocation structure of the samples in initial state; (b) morphology picture in the contact area (*a*: ×30, *b*: ×40).

clusions under micro-indentation [6, 7]; formation and healing the cracks and micropores [8–13], etc. Certain success was achieved in studying the role of fluctuation mechanism for deformation and destruction of solids [14–17].

In the works [18] and [19] the formation kinetics of the contact zone between squeezed solids at high temperature was studied and it was shown that the transfer processes are realized by the dislocation-diffusion mechanism (DDM) in this case.

In the present work, the results on studying the DS formation kinetics in the contact area under squeezing the solids, its geometry and evolution are given for simulation experiments with KCl single crystals.

2. Experimental

Evolution of DS during formation of the contact was investigated in simulation experiments with samples in the shape of single wedges and combs of wedges with vertex angle 2α in the range from 60° to 120° and with the radius of curving the wedge vertex — of an order of several hundreds of microns. The samples were prepared from a KCl single crystal properly annealed at T = 730°C (for 10 h). From the single crystal block, the rough samples of (10×10×6) mm^3 were cut off, and then they were polished to obtain the required geometry shape using wet polishers in order to avoid substantial force impact. The prepared samples were repeatedly annealed at T = 730° during 0.5 h for additional normalization and dehydration of the surface. The density of dislocations (DD) in the samples was $(1\div2)\cdot10^5$ cm^{-2} (Fig. 1a).

The wedge vertex had ⟨100⟩ type direction and was clamped to the substrate {100} type plane parallel to ⟨100⟩ type direction. The experimental temperatures were T_{room} and T = 550°C ($\approx0.8T_m$). The main annealings were for 5; 15; 90; 240 and 480 min. At $T = T_{room}$, the loading was carried out or 5 min through a molybdenum wire with diameter 0.5 mm. The load was varied in the range where the optically resolved DS could be obtained in order to observe its configuration and formation kinetics. At room temperature, the load was from 20 to 800 N/m, and in high temperature experiments it was 800 N/m.

3. Results and discussion

Mechanical stress in the contact area of the solids "wedge-half-space" is most suitable to describe using the solution of the 'plane problem' where the concentrated and uniformly distributed along the wedge vertex load P (N/m) acts in the direction of the bisecting line for the vertex angle 2α (Fig. 2). This solution suitable both for the wedge and for the half-space is given in [20]. In the cylindrical coordinates (r, θ) with a center in O point, the stress tensor is:

$$\sigma_r = \frac{kP\cos\theta}{r}; \quad \sigma_\theta = \sigma_{r\theta} = 0, \tag{1}$$

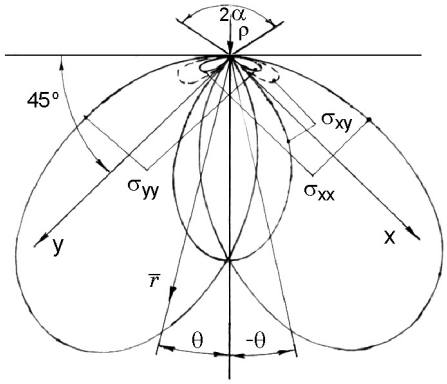


Fig. 2. Normal and tangential stress distributed in the crystal.

where $k = \frac{1}{\alpha + (1/2)\sin 2\alpha} = \frac{2}{2\alpha + \sin 2\alpha}$. Taking into consideration the fact that in KCl type single crystals, the dislocation glide systems include the {110} type planes and ⟨110⟩ type directions, let us take the {100} planes as coordinate ones with origin in O point, and then superpose one of the planes with upper boundary of the half-space, and the second one coinciding with (*XOY*) plane — in the perpendicular direction. The axes *OX* and *OY* we directed along ⟨110⟩ type crystallographic directions (Fig. 2). The stress tensor within these axes becomes:

$$\sigma_{xx} = \frac{kP}{2r}(\cos\theta(1 - \sin 2\theta));$$
$$\sigma_{yy} = \frac{kP}{2r}(\cos\theta(1 + \sin 2\theta)); \qquad (2)$$
$$\sigma_{xy} = \frac{kP}{2r}(\cos\theta \cdot \cos 2\theta).$$

The distribution of shear and normal stresses depending on θ given by Eq.(2) for $r = r_{const}$ (the stress distribution diagram in polar coordinates) is shown in Fig. 2. All the stress components (σ) decrease with distance (*r*) according to $\sigma \sim 1/r$. The value of *k* factor in (1) and (2) increases as the wedge vertex angle 2α decreases. For the half-space ($2\alpha = \pi$), the value $k = 2/\pi \approx 0.64$. For the wedge with $2\alpha = \pi/3$, the value *k* 1.045, i.e. the stresses in the wedge with $2\alpha = \pi/3$ are by a factor 1.5 higher than in the half-space, onto which the wedge affects. Nevertheless, being clamped to the half-space, the wedge edge is practically not deformed, while in the half-space the groove-like cavity occurs due to embedding the wedge. Such result is not unexpected, because the Eqs. (1) and (2) are valid for the case of linear contact. For the case of real solids contacting, for example, for spherical or cylindrical shaped ones, the problem is solved by Hertz. It was shown that in the contact, both solids are deformed, and the contact surface forms, so, it is possible to calculate its geometric parameters and stresses in the points of the surface. If the second solid is flat, the flat surface can only flex under loading transmitted by the first (spherical or cylindrical) solid. The contact surface occurs in the form of a crater or a groove. The solution we use, strictly speaking, is valid beyond the bounds of the contact surface. If the plastic zone also occurs, the Eqs.(1, 2) are not accurate in this zone. Beyond the bounds of the plastic zone, (1) and (2) describe correctly the pattern of the stress state [20].

Under conditions of the wedge loading, due to higher stresses in the wedge and limitation of its space by lateral surfaces, the dislocation density increases with advancing rate. In the wedge, a certain level of strain hardening is achieved quickly, and the fixed DS configuration remains almost unchanged in comparison with dynamics of DS variation in the half-space, where the main portion of the substance squeezed out by the wedge is carried away. In this connection, the series of micrographs is shown below mainly for dislocation structures in the half-space. To the end of the experiment, the dislocation configurations are practically similar both in the half-space and in the wedge with somewhat higher dislocation density in the wedge (Fig. 3d).

Affect of a sharp flat wedge (limited by flat intersecting surfaces), which edge is parallel to ⟨100⟩ type crystallographic direction, onto the {100} type plane can be considered approximately as micro-indentation of the crystal with a concentrated force. Therefore, it can be expected that under loading at room temperature, dislocation "rays" (DR) should occur in ⟨110⟩ directions from the origin of force (as under micro-indentation), that is observed indeed (Fig. 3a). The "rays" are somewhat blurred that is connected with the indenter "linearity" and unavoidable superposition of DSs (in the plane where the DS is observed) from the neighboring points of the linear indenter.

The mechanism of DR formation is as follows. The shear loops generated on each side of the planes $x = 0$ and $y = 0$ have different signs caused by difference of σ_{xy}

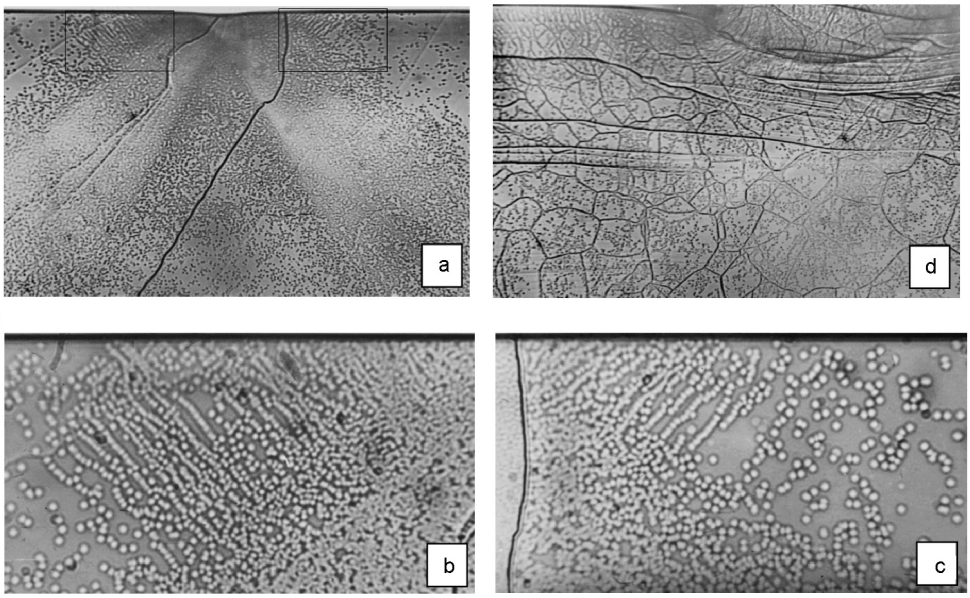


Fig. 3. Dislocation structures: *a*, *b* and *c* correspond to conditions $P = 200$ N/m^2, T_{room} and annealing time $t = 5$ min; *a*(×150); *b* and *c* are fragments of Fig. 2a (×600); *d* corresponds to conditions $P = 800$ N/m^2, $T = 550$°C and annealing time $t = 10$ min (×280).

signs on each side of these planes (Fig. 2). The edge portions of such shear loops also have different signs, therefore in total, they are equivalent to motion of interstitial prismatic loops from the contact area, which transfer the substance from the contact area into bulk of the crystal.

A part of this substance is squeezed out onto the surface near the contact. The volume of this substance is insignificant and amounts (by estimations made using the sample surface profile analysis after loading [19]) less than 10 % of the total squeezed volume. From Fig. 2, it can be seen the schematic model and the reason of the substance squeezing. In sub-surface layers in the small neighborhood of the contact surface, the substance is compressed, thus has a "trend" to partial squeezing onto the closely set surface. In Figs. 3b and 3c the fragments of micrographs for DS from Fig. 3a are shown, where the slip bands from the "rays" to the crystal surface as a result of sliding the squeezed substance to the surface. Note, that in the formation of piles and DRs, also the crowdion mechanism can take part. This is connected with high inhomogeneity of strain in the contact area due to the local character of force impact (Fig. 1b) and high gradients of strain in the contact area [21]. The symptoms of the crowdion mechanism participation under indentation were observed in [6, 7], by the authors [13], and in the works by V.N.Rozhanskiy et al. As the temperature increases, the general scheme of the substance transport caused by the stress distribution remains, but the role of thermal-activated processes raises quickly, which influence on the character of motion of plasticity carriers and the substance (dislocations and point defects). At 550°C we already do not observe marked DR going from the wedge vertex. As it is seen from Fig. 4a, b, c, d, after annealing under loading, the contact zone is occupied by dislocations in the limits of some area rather uniformly on a macro-scale, with decreasing density as moving away from the contact. The formation of slip bands in the {110} planes under deformation continues in principle. But the formed flat assemblages of dislocations which remained at T_{room} generating DR, now lose stability because of lost stability of the flat assemblages (screw portions of dislocations, for which all the crystallographic planes may be slip planes). The screw components of the loops realize transversal sliding away from DR. At that, short portions of "sitting" edge dislocations are formed which are new Frank-Read sources. By the repeating transversal sliding the screw dislocation components create new shear loops outside the DR. Going out the screw portions from the flat assemblages and creation of new Frank-Read sources results in somewhat weakening of the disloca-

tion assemblage as a whole, generation of new dislocation loops, and continuation of the contact formation process. Such expansion of the dislocation assemblage termed as double transversal slip (DTS) is the known phenomenon [22] and was often observed. The expansion of the "rays" by DTS mechanism begins from the end lateral bands. The dislocations occupy the half-space up to significant distances from the contact place both by width and depth.

In the middle of the crystal below P force, there is a zone free of the traces of plastic deformation, with low DD (Fig. 4d), which is caused by the stress state character (Fig. 2). At high temperature, the piles are formed by the same mechanisms as for the room temperature case, and additionally the diffusion mechanism engages.

The consecutive theoretical analysis of DS development under conditions of plastic deformation taking into account not only external conditions, but also the properties of DS itself [1–5, 23, 24] has shown existence of some correlation interaction between dislocations. This gives a negative addition to energy of the dislocation system, thus always provokes an effect of attraction between the dislocations. Under conditions of the correlation interaction, a certain dynamics of fluctuations occurs, and as DD (ρ) increases, a bifurcation point is achieved. At $\rho = \rho_c$ (ρ_c is critical density), the homogeneous state becomes stratified, and cells with L_c size are formed. The type of the formed cellular structure (CS) is determined by a set of sustained collective excitations (modes) termed as parameters of order. In [4] the cases are analyzed for the number of the modes N not more than 5. The analysis shows that at $N = 1$, a single-dimensional periodic structure forms; at $N = 2$ and $N = 3$, respectively, rhombic and hexagonal ones form; and at $N = 4$ and $N = 5$, more complicated structures with internal symmetry in the cells occur.

In [5] the expressions for ρ_c and L_c are obtained in an explicit form:

$$\rho_0 > \rho_c = \frac{8\pi e(1-\nu)^2}{b^2}\left(\frac{\sigma_p}{G}\right)^2, \qquad (3)$$

where σ_p is Peierls threshold, G is shear modulus, b is Burgers vector, and ν is Poisson coefficient,

$$L_c = \sqrt{8\pi^2/e(e-1)} \cdot \rho_0^{-1/2}, \qquad (4)$$

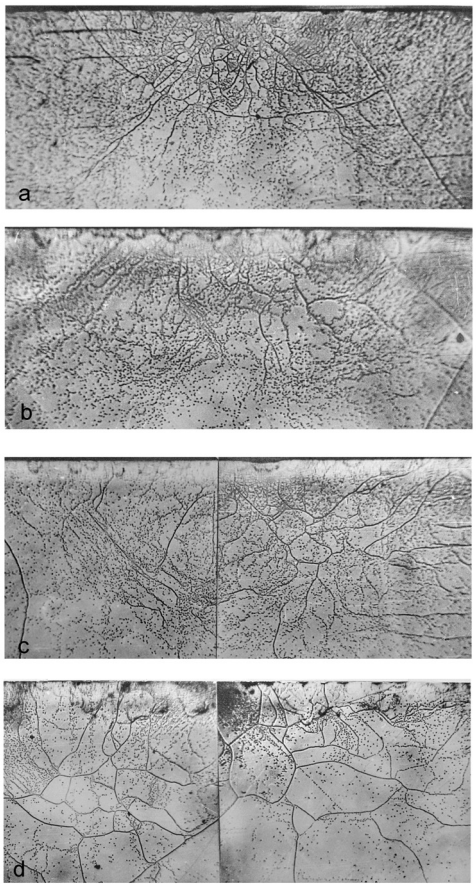


Fig. 4. Dislocation structures (×20): *a*, *b*, *c*, and *d* correspond to conditions $P = 800$ N/m^2, $T = 550$°C, for annealing times t (min.): *a* — 5, *b* — 15, *c* — 90, *d* — 480.

where ρ_0 is true dislocation density in their homogeneous state.

Comparison of experimental data with theoretical analysis results indicates the qualitative confirmation of (4): as moving away from the contact, homogeneous dislocation density drops, and the cell size increases. The tendency to the cell formation in DS is observed experimentally even in the earliest stages of annealing, that does not allow to determine exactly the DD (ρ_0) in homogeneous state. Thus, we solve an inverse problem of ρ_0 determination by the observed L_c value. According to Figs. 4a and 3d, after annealing under load for 5 min, the cell size near contact is $L_c \approx 5 \cdot 10^{-3}$ cm. Substituting L_c into (4) gives: $\rho_0 \simeq 10^7$ cm^{-2}.

Also the residual DD (ρ_{rem}) in the cell can be determined from Fig. 3d. In the sample annealed under the same conditions during 10 min ρ_{rem} amounts on average

$(5\div8)\cdot10^6$ cm^{-2} (Fig. 3d) that is close to the ρ_0 estimation by L_c value (10^7 cm^{-2}). The real DD in homogeneous state can be estimated more precisely only in low-temperature experiments. From the pictures like Fig. 3b, c, it can be obtained that near the contact (at the limit of resolution, Fig. 2b, c) $\rho \simeq 2.5\cdot10^7$ cm^{-2}. Below (Fig. 2a, between "rays"), $\rho \simeq 2.5\cdot10^6$ cm^{-2}.

Using Eq.(3) we estimate the critical DD value (ρ_c) necessary for CS formation. Substituting in (3) the correspondent values and taking into account that possible ratios σ_p/G are approximately in the range of $10^{-5}<(\sigma_p/G)<10^{-3}$, $b = 3\cdot10^{-10}$ m, and $\nu \simeq 0.3$ we obtain $10^6<\rho_c<10^{10}$ (cm^{-2}). The given above experimental ρ_c estimations satisfy this inequation indicating the developed in [4, 5, 23, 24] theoretical concepts agree with our experimental results.

As it is seen from micrographs (Fig. 3), during annealing the cells increase their sizes significantly with time (after 8 h of annealing — by an order of value) achieving the order of $5\cdot10^{-2}$ cm and even up to 10^{-1} cm sizes. If the growth of the cells is thought as a result of diffusion, we can use the known relation from diffusion theory $L^2 \simeq Dt$ (L is distance of substance transfer, D is diffusion coefficient, and t is diffusion time) and evaluate the diffusion coefficient by our observed data. Substituting $L = 5\cdot10^{-2}$ cm, and $t = 2.88\cdot10^4$ s we obtain $D \simeq 8.7\cdot10^{-8}$ cm^2/s. This value is somewhat overestimated. Therefore, it may be assumed that the cell growth realizes both by dislocation slip mechanism and by diffusion climb.

As to CS symmetry and its crystal likeness, it may be said the following. The observed CS (Fig. 3d, 4d, etc.), firstly, cannot be classified as one-dimensional periodic one, so in the terms of the developed in [4] theory this contains the number of parameters of order obviously $N>1$. On the other hand, the cells contain no internal elements assigning their intrinsic symmetry of rotation, that signifies according to the theory that the number of the order parameters $N<4$, i.e. really $N \simeq 2$ or 3. At these N, as it is noticed in [4], a crystal-like cellar structure should be formed, respectively, of rhombic or hexagonal type. It is difficult to insist that we observe one of these types. But we indeed observe polygonal cells, as a rule, of not right form with different number of sides (but, usually, 4÷6 sides; see Fig. 3d, 4d).

This is the structure of a mixed type with rhombic and hexagonal cells (rarely triangle cells observed as well as more than six-sided ones). In this connection, it is worth to note an important obstacle that we observe plastic deformation in the local area near the stress concentrator, high and rather inhomogeneous strain with large gradients that is well seen in the picture of the contact area morphology (Fig. 1b). Near the contact surface, high dislocation occupation, high concentration of point defects, and disintegration of single crystal occur. In our experiments, nearby the contact area the block off-orientation estimated by blurring the spots on inverse Laue-grams achieves 5÷10 degrees. In fact, it is a low bound where a fragmentary structure forms [1–3].

A number of phenomena are known, which have behavior peculiarities under conditions of high strain inhomogeneity. There is, for example, information [25] that under local conditions, dynamical crystallization was observed as well as small-angle and high-angle grain boundaries, micropores and micro-cracks, etc. These notes may serve a basis for assumption that the mentioned conditions for localized deformation can affect the CS form. Taking this fact into account, it may be assumed that the given experimental data and estimations are enough to consider these as supporting qualitatively the theoretical predictions [4, 5, 23, 24].

The obtained knowledge on the regularities and kinetics of DC formation under plastic deformation in local area nearby the contact for the samples of KCl single crystals is in full measure valid for experiments with other single- and polycrystalline materials, including copper samples studied in [19]; the fact signifies about validity and effectiveness of the developed in [18, 19] dislocation-diffusion mechanism (DDM) for formation of the contact between squeezed crystalline solids. Let us pay attention to the follows. In the kinetic curves $S(t)$ obtained in [19] (S is relative width of the contact of W/W_0) after $t_{st} \simeq 5\div10$ min from the beginning of the process, an abrupt retarding the contact growth occurs that is observed as changing the behavior ("knee") of curves; this was explained as the fact that t_{st} is the formation time of quasi-stationary dislocation assemblage locking activity of a dislocation source on the surface of the contact. The thermal-fluctuation de-

parture of dislocations from the "tail" of the assemblage opens the source and controls the process further. This is the moment when the transition from dislocation mechanism to DDM of the contact formation takes place. By the behavior of observed kinetics of DS variation it is also seen that after $t_{st} \simeq 5 \div 10$ min the DS looks like completely formed (quasi-stationary) (Fig. 3d, 4a, d). Thus, actually postulated in [19] by the "knee" of kinetic curves, quasi-stationary of the dislocation assemblage after t_{st}, in our experiments is supported by the kinetics of DS variation.

At $t > t_{st}$, only the ordering of the cellar structure and its enlarging is observed along with gradual decreasing the dislocation background density, that takes place due to both dislocation annihilation and embedding these into inter-cell walls. However, after the maximum annealing time (8 h in our experiments), relative disorientation of cells remained insignificant (excluding the area adjacent to the contact surface). Observed in Fig. 4d several slip bands intersecting a few cells not far from the contact do not show visible 'knees' at the cell boundaries, therefore the structure remains cellar and does not became fragmentary [1–3] which is characteristic for large deformations.

4. Conclusions

Evolution of dislocation structure during the contact area formation under loading was studied in the model experiments "wedge-plane" for KCl single crystal at room and high temperatures $0.8T_m$. The existence of the postulated earlier quasi-stationary dislocation assemblage was supported, with which formation the rapid stage of the dislocation mechanism for contact formation is finished. It was shown on the basis of DS observations and analyzing the stress distribution field nearby the contact, the main mechanisms of substance transport away from the contact into bulk of the crystal as well as partial squeezing this onto the surface are dislocation mechanism and DDM. In connection of locality of the force impact and, as a consequence, high strain gradients, also participation of the crowdion mechanism is possible. It was established that as the DD increases and achieves the critical value (ρ_c), the dislocation structure became cellar. The cell size L_c increases with DD decreasing in homogeneous state in accordance with the theory. By experimental observations the DD critical value (ρ_c) at which the CS formed was estimated. This was found to be of the order of 10^7 cm^{-2}. This value is in the range of theoretically predicted ρ_c values, at which the CS formation should be expected: $10^6 < \rho_c < 10^{10}$(cm^{-2}).

The crystal-likeness of CS followed from the theoretical analysis is supported qualitatively. The CS observed consists of cells-polygons with the number of sides usually from 4 to 6, i.e. this looks like a mixture of cells with rhombic and hexagonal symmetry, as predicted by the theory.

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Кінетика формування дислокаційної структури у приконтактній області здавлюваних кристалічних тіл

Ю.І.Бойко, М.А.Волосюк, В.Г.Кононенко

Експериментально досліджено еволюцію дислокаційної структури при формуванні контакту у модельних експериментах "клин-площина" з монокристала KCl під навантаженням при кімнатній і високій ($0.8T_m$) температурах. Показано, що на першій (швидкій) стадії процесу за рахунок емісії дислокацій формується квазістаціонарне дислокаційне скупчення. Встановлено, що при високій температурі ($0.8T_m$) комірчаста структура (КС) формується на найраніших етапах відпалу під навантаженням. Експериментально визначено критичну щільність дислокацій ρ_c, при якій КС формується: $\rho_c \approx 10^7$ см$^{-2}$, що за порядком величини відповідає її розрахунковому значенню ($10^6 < \rho_c < 10^{10}$) см$^{-2}$. Кристалоподібність КС, що передбачається теорією, якісно підтверджується; КС містить суміш комірок — багатокутників з числом сторін переважно від 4 до 6.