

Peculiarities of high-temperature plastic straining anisotropy in with NaCl type single crystals in creep conditions

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The peculiarities of high-temperature creep anisotropy of NaCl and KCl single crystals have been studied under two stressed states. The relationship between the number of active easy slip systems and the straining rate has been established. The conditions of dislocation reactions in NaCl type single crystals and the results of dislocation interactions have been considered.

Исследованы особенности анизотропии высокотемпературной ползучести монокристаллов NaCl и KCl при двух видах напряженного состояния. Установлена связь между числом действующих систем легкого скольжения и скоростью деформации. Проанализированы условия прохождения дислокационных реакций в кристаллах типа NaCl и результаты взаимодействия дислокаций.

In the course of service, the single crystals are subjected to mechanical stresses. The functional properties of the crystals may undergo variations due to changes in the crystal shape and dimensions. To forecast the single crystal behavior, reliable information on the plastic straining thereof are necessary. At a specific temperature and the external stress level, the straining rate is known to depend on the initial dislocation structure of the single crystal, its crystallographic features (the number of possible easy slip planes), and the stressed state type [1]. The influence of the latter two factors can be elucidated by studying the plastic strain anisotropy of the single crystals at various orientations thereof relative to the external stresses. This study is simplest to carry out for crystal structures with a limited number of the easy slip planes, e.g., for f.c.p. metal single crystals [1, 2] where there is a single preferred easy slip system. For crystals with larger numbers of easy slip systems (f.c.c and b.c.c. lattices, NaCl type alkali halide crystals), it is much more difficult to study and analyze the plastic strain anisotropy. In those cases, not only the crystal orientation with respect

to the external force but also the stressed state type it to be varied.

The experimental works on the orientation-depended plastic straining of metal single crystals are known in literature. Those studies were carried out mainly at low temperatures (ranging from liquid helium to room ones) in the active loading mode. In contrast, there are essentially no experimental data on the plasticity anisotropy for alkali halide crystals of CsCl and NaCl lattice types, especially at high temperatures. Such data, however, are necessary to develop a modern plasticity theory and to forecast the behavior of devices based on those crystals (in particular, scintillators) in high-temperature service conditions under external mechanical stresses. Only few works are known dedicated to the high-temperature anisotropy of the CsI crystal plasticity in the active loading mode [3] and in the course of creep [4]. In this work, presented are the results of experimental investigations in the orientation-depended high-temperature (up to $T \approx 0.95T_m$) plastic strain in the creep mode for alkali halide crystals of NaCl type, namely, for annealed

NaCl and KCl single crystals with the initial density of chaotically distributed dislocations $\rho_0 \approx 5 \cdot 10^9 \text{ m}^{-2}$ and the average block size $L \approx 10^{-3} \text{ m}$.

The plastic straining rate is defined by the number of mobile dislocations involved in the process and by the speed thereof, v . The v value depends very heavily on the level of the effective shear stresses $\tau_{eff} = \tau - \tau_i$, where τ denotes the reduced shear stresses in the dislocation slip plane along the corresponding Burgers vectors caused by the external stresses σ ; τ_i , the reverse internal stresses hindering the dislocation slipping. As a result, in the crystals containing several physically equivalent dislocations, under pre-specified external stresses σ , the dislocation begin to raise and move in the planes where the orientation factor α (that defines the shear stress value τ) is the largest. $\tau = \alpha\sigma$, $\alpha = \cos\varphi\cos\theta$, φ being the angle between the direction of the external force \mathbf{F} and the Burgers vector of the dislocation; θ , the angle between \mathbf{F} and the normal to the slip plane. The effectiveness of dislocations as the plastic strain carriers can be influenced, the external stress σ value being pre-specified, by varying the reduced shear stress value τ (by orienting the single crystal with respect to the external load) and the number of active dislocations for which τ is the largest (by varying the ratio of the crystal geometric dimensions under uniaxial load). In our studies, two straining methods were used: uniaxial compression (the stresses are homogeneous over the sample cross-section) and pure bending when the stress value and sign vary over the cross-section. In all the cases, the external stress value was sufficient to form new dislocations in the crystal ($0.5 \text{ MPa} \leq \sigma \leq 0.8 \text{ MPa}$). The dislocation structures in the crystals subjected to straining at high temperatures were compared to those in the crystals strained in similar modes at room temperature and then annealed at the same elevated temperatures in the absence of external loading.

In NaCl crystals, it is just $\langle 110 \rangle \{110\}$ that is the easy slip system, i.e. 12 physically and geometrically equivalent edge dislocations are present. To study the creep anisotropy under uniaxial compression, a single crystal shaped as a parallelepiped of the height h and the cross-section $a \times c$ was subjected to loading in two directions, $\langle 100 \rangle$ and $\langle 111 \rangle$. In the first case, the orientation factor $\alpha = 0.5$ in four planes,

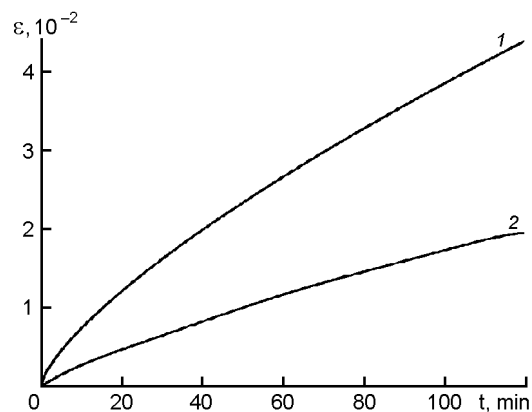


Fig. 1. Time dependence of strain. $T = 600^\circ\text{C}$, $\sigma = 0.8 \text{ MPa}$. Crystal orientation: $\langle 100 \rangle$ (1), $\langle 111 \rangle$ (2).

while in the second one, $\alpha = 0$ in all 6 planes of the $\{110\}$ type. Therefore, it is natural that the creep speed $\dot{\epsilon}_{100} > \dot{\epsilon}_{111}$ (Fig. 1) when the dislocations in the easy slip planes are immobile. At high temperatures, those crystals could be strained in diffusion mode. However, the creep speed $\dot{\epsilon}_{111}$ observed in experiment exceeds by several orders that estimated from the diffusion mechanisms. Therefore, it can be concluded that in NaCl single crystals, the $\langle 110 \rangle \{100\}$ system starts to act when the dislocation formation and displacement in the $\langle 110 \rangle \{110\}$ easy slip systems is impossible.

Gilman has shown in one of earlier works [5] aimed at the study of plastic straining under active loading that when LiF crystals pricked out along the cleavage planes are twisted, the dislocations arise and move really along $\langle 110 \rangle$ directions in $\{100\}$ planes. In this case, the yield limit exceeded almost by a decimal order that found at straining in the easy slip system. A similar situation seems to take place at high-temperature creeping of $\langle 111 \rangle$ oriented NaCl single crystals in our experiments.

The number of active easy slip systems in the single crystals being compressed in the $\langle 100 \rangle$ direction can be changed by varying the geometric dimension ratio of the put-out rectangular samples. As the cross-section is square ($a \approx c$), four $\{110\}$ type planes are equivalent and eight various dislocations may contribute to the strain (Fig. 2). In Fig. 2, the uniaxial stress σ is applied along the OZ axis ($[001]$ direction) to the XOY plane. At rectangular cross-section ($a \approx 2c$), the dislocation arise and move predominantly only in two mutually perpendicular planes. If the crystal cross-section

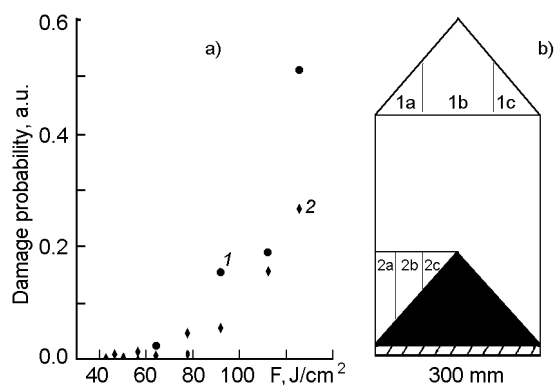


Fig. 2. Crystal straining scheme under compression along the OZ [001] axis.

minimum dimension (c) is parallel to the OY axis, the only four dislocations \mathbf{b}_1' , \mathbf{b}_2' , \mathbf{b}_3 , and \mathbf{b}_4 are involved in the plastic straining (Fig. 2). This fact can be explained by that the strain is effectively contributed to by the dislocations in equivalent easy slip planes (with the same τ value) which need to overcome less hindrances in the motion. So, if the stoppers are distributed homogeneously over the crystal volume, the shorter is the way to be passed by a dislocation to the surface, the easier is its slipping and the more it will contribute to the strain. This explains also the increasing creep speed observed in our experiments as the minimum transversal dimension (c) of the crystal diminishes (Fig. 3, curves 1 and 2).

It could be expected that, at the same c values, the crystal with square section where 4 slip planes are active (Fig. 2) would be strained faster than that of the rectangular section. However, the experiments have shown that, if $a \approx 2c$, the creep speed is higher than for crystals with $a \approx c$ (Fig. 3, curves 2 and 3). The creep of the crystals with square section is characterized as a rule by a poor reproducibility of the results. Perhaps this is caused by the fact that, at the straining in four planes, the slipping is more active in two mutually perpendicular planes for certain reasons (skew loading, the crystal structure inhomogeneities, etc.), the slip in two other planes becomes suppressed. Thus, an increased number of the slip planes having the same value of the reduced shear stress does not result in obligatory increase of the plastic straining rate. The dislocation interaction in the course of straining that may stop the dislocations should be taken into account.

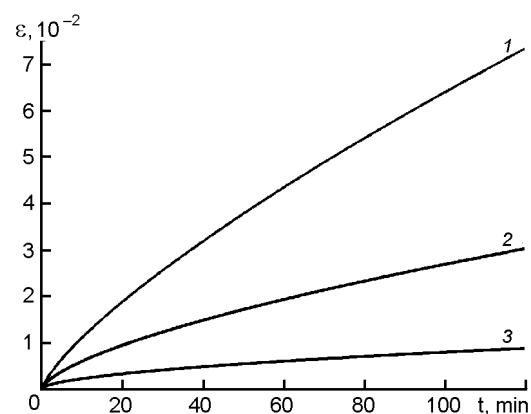


Fig. 3. Time dependence of strain for $\langle 100 \rangle$ oriented NaCl crystals. $T = 600^\circ\text{C}$, $\sigma = 0.8 \text{ MPa}$. The samples with rectangular (1, 2) and square (3) cross-section. $l_{\min}(1) < l_{\min}(2) \approx l_{\min}(3)$.

The reduced dislocation mobility may be due to several causes. First, the moving dislocation may be stopped by the impurity atoms. Second, the stopping may occur at crossing of the forest dislocations. Third, it may be due to interaction with the elastic stress fields of other dislocations, in first turn, with the dislocations of the same slip plane that are stopped by some hindrance. The fourth reason for reduction of the number of mobile dislocations consists in dislocation reactions resulting in formation of sessile dislocations. The first two possibilities are the same for all dislocations. The third cause results in an increased level of reverse internal stresses and, as is mentioned above, the stopping probability increases with increasing path length to the crystal surface (enlarged crystal dimensions). In our opinion, it is just the fourth possibility forms the main reason for reduced number of mobile dislocations at the high-temperature plastic straining.

Let the dislocation reactions in the NaCl type crystals be considered in more detail. Two dislocations with Burgers vectors \mathbf{b}_1 and \mathbf{b}_2 can react with one another and form a dislocation with the Burgers vector \mathbf{b} if the Frank criterion [6] $|\mathbf{b}_1|^2 + |\mathbf{b}_2|^2 \geq |\mathbf{b}|^2$ is met. The inequality means that the reaction results in a reduced crystal energy, i.e. in relaxation of internal stresses. The NaCl type crystals contain 6 easy slip systems, thus, there are 12 dislocation types with different Burgers vectors. Therefore, 66 various dislocation reactions are possible. Let us consider as an example the possible dislocation reactions of a rectilinear edge dislocation having the Burgers vector $\mathbf{b}_1 = a/2[\text{T}01]$ and a vector tangent to the

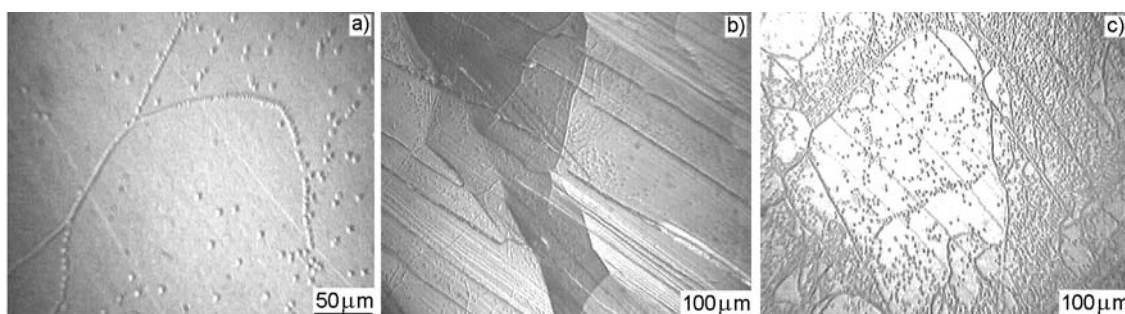


Fig. 4. Dislocation structures of $\langle 111 \rangle$ oriented NaCl crystals strained at room temperature and then annealed (a) and after creep (b, c). $T = 600^\circ\text{C}$, $\sigma = 0.8 \text{ MPa}$.

dislocation line $\mathbf{l}_1 = [100]$ with other dislocations of the crystal:

$$\frac{a}{2}[\bar{1}01] + \frac{a}{2}[10\bar{1}] = 0, \quad (1)$$

$$\frac{a}{2}[\bar{1}01] + \frac{a}{2}[\bar{1}\bar{1}0] = \frac{a}{2}[\bar{2}11], \quad (2)$$

$$\frac{a}{2}[\bar{1}01] + \frac{a}{2}[101] = a[001], \quad (3)$$

$$\frac{a}{2}[\bar{1}01] + \frac{a}{2}[01\bar{1}] = \frac{a}{2}[\bar{1}10]. \quad (4)$$

The first reaction results in annihilation of the dislocations. The second one is impossible because $b_1^2 + b_2^2 < b^2$ (there are 4 reactions of that type). The third reaction does not provide a gain in the crystal energy, that is, it may occur under an external action but not spontaneously. As a result, a stiff sessile dislocation is formed. Interactions of that type may take place in crystals with rectangular cross-section (there are 2 reactions of that type). The experimental proofs for reality of those reactions are very few [1, 6]. The fourth dislocation reaction is energy-favorable and may occur spontaneously or under an insignificant external action, since $b_1^2 + b_2^2 > b^2$. As a result, a sessile dislocation with the tangent vector $\mathbf{l} = [111]$ is formed. This type reactions occur in the crystals with square cross-section (Fig. 2, upper left drawing). There are 4 reactions of that type.

The dislocation structures of the $\langle 100 \rangle$ oriented crystals are qualitative similar to each other in the samples strained by compression at room temperature with subsequent annealing and those tested in the high-temperature creep mode. Those are characterized by a typical small-block structure with a network of dislocation boundaries containing inside it both single chaotically arranged dislocations and suspended boundaries with

one or both ends broken inside the blocks [7]. Both rectangular and square cross-section crystals take a barrel-like shape after creep.

The $\langle 111 \rangle$ oriented crystals are very difficult to be strained at room temperature. After the high-temperature annealing, the dislocation structure of these crystals differs considerably from that of the $\langle 100 \rangle$ oriented ones. The block structure is not broken due to insignificant preliminary straining. However, in some crystal areas, unusually configured accumulations of dislocations appear (Fig. 4a), namely, sections of the usual dislocation boundaries passing into very curved scraps of boundaries being in building. There is an impression that the straining at room temperature occurred in local areas of the crystal. The dislocations formed in the $\langle 110 \rangle \{100\}$ system take the diffusional mobility at high temperatures and interact with each other, so tending to form configurations with lower energy as compared to that of the ensemble consisting of single dislocations. However, the boundary building process is slow, because the slip starting stresses are considerably higher than in the $\langle 110 \rangle \{110\}$ system. This is evidenced by repeated anneals of those crystals.

The structure of $\langle 111 \rangle$ oriented crystals after the high-temperature plastic straining in the creep mode differs considerably from that described above. Two characteristic features are to be noted. First, some areas of the crystal undergo a macroscale reorientation (Fig. 4b). The misorientation angles of the crystal fragments amount to several degrees, i.e., the single crystal transits into a polycrystal in some areas. This evidences that the plastic straining involving the slipping dislocations is hindered even at high temperatures. Second, the usual dislocation boundaries between the blocks are formed as in the creeping $\langle 100 \rangle$ oriented crystals, but considerable areas of the crystal contain a

high density of single dislocations (Fig. 4c). Such a structure remains essentially unchanged under subsequent high-temperature anneals. This structure is similar to that formed under annealing of single-crystal NaCl plates preliminarily bent at room temperature with the bending force applied to the $\{110\}$ plane [8]. The post-annealing dislocations have been found to be sessile ones [8]. The formation thereof results from the dislocation reactions of the (4) type when the dislocation move in five $\langle 110 \rangle \{110\}$ easy slip systems under the preliminary straining.

The strain in these system is, however, forbidden at the creep of the $\langle 111 \rangle$ crystals. What is the origin of those dislocations? In our opinion, there are two possible causes. First, in the course of plastic straining in the $\{100\}$ planes, the dislocations meet each other, so dislocation reactions are possible. The $\langle 110 \rangle \{100\}$ system contains also 12 physically equivalent edge dislocations. As the analyze shows, at the straining in the $[111]$ direction, the maximum orientation factor is $\alpha \approx 0.47$ for 6 dislocations that may be formed and move; as a result, 15 dislocation reactions are possible. Only 6 thereof may result in a reduced internal energy of the crystal. An example is $a/2[110] + a/2[101] = a/2[011]$. Other reactions are similar to the exemplified one. Those reactions result in formation of pure edge dislocations with lines lying in three directions of the $\langle 100 \rangle$ type and the slip planes of the $\{110\}$ type. This means that the dislocations are formed in the easy slip system. As the strain increases, the slip planes are turned [2] so that the angle between the crystal axis and the active slip systems becomes diminished. The crystal orientation changes in the active straining areas causes the motion of the dislocations. The second (and perhaps the main) cause of the dislocation formation in the easy slip system consists in that, as a result of the crystal re-orientation due to straining, the orientation factor α in the easy slip systems may become nonzero, thus, dislocations appear in those systems. The interaction between those dislocation causes the formation of numerous but local accumulations of sessile dislocations that are found in experiment (Fig. 4c).

The sessile dislocations occupying a considerable fraction of the crystal volume are formed at bending of NaCl type single crystals oriented in a specific manner. If the single-crystal plates pricked out along the $\{100\}$ cleavage plane are bent around the

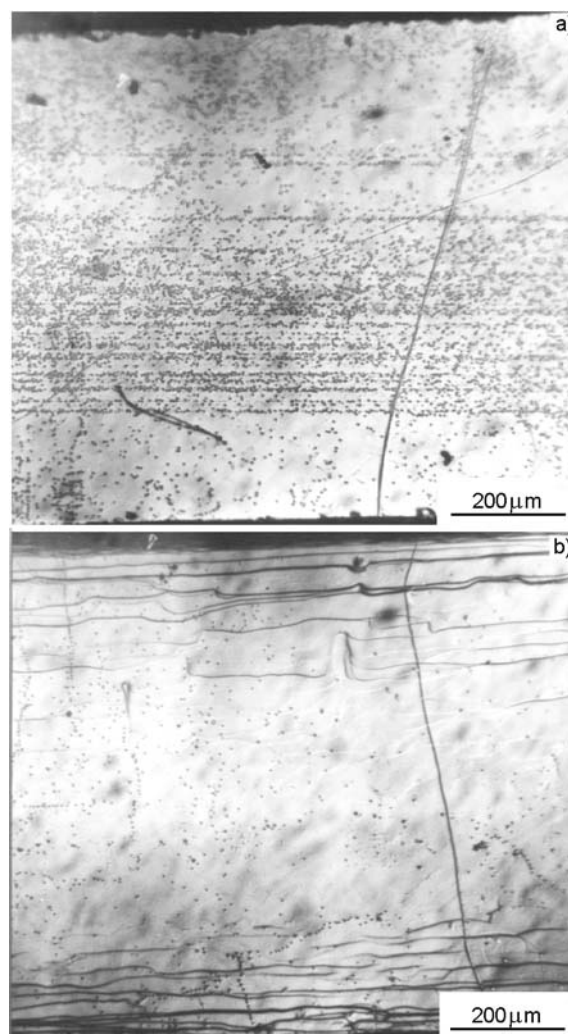


Fig. 5. Structures of the same area in the crystal bent at room temperature (a) and annealed (b).

$\langle 100 \rangle$ direction and then annealed, the dislocation structure is a well-known [9, 10] system of polygonal boundaries with single dislocations arranged chaotically. The single-crystal plates cut out so that the bending load is applied to the $\{110\}$ planes are much more difficult to strain than pricked out ones. When such single crystal are annealed, ordered ensembles of sessile dislocations are formed in the whole strained area [8]. The high-temperature bending of those crystals under a constant external loading (in the creep mode) is also hindered as compared to the pricked-out plates, and the dislocation structures thereof exhibit the ensembles of sessile dislocations over the whole volume.

As a (110) oriented crystal is bent, the stresses are found to be distributed [11] in such a manner that the highest tangential stresses act in one (110) plane located in the plate depth, in the middle part thereof. In

four planes (011) , $(0\bar{1}\bar{1})$, (101) , $(10\bar{1})$, the maximum tangential stresses are almost one order lower than those in the (110) plane parallel to the surface to which the external force is applied. The hindered plastic straining of the crystals oriented as mentioned above seems to be due to two causes. First, the dislocations in the (110) plane are generated in the crystal volume (Fig. 5a) and not at the surface thereof where the dislocation are formed easier as a rule. A specific feature of the slip lines (Fig. 5a) consists in that dislocations with anti-parallel Burgers vectors (i.e. dislocation loops) are present therein in essentially the same numbers. We made sure this fact when studying and comparing the dislocation structures after a small bending strain, when the dislocations are still not arisen in the four slip planes with low shear stress level (Fig. 5a), and those after subsequent annealing without external stresses (Fig. 5b). It is seen in Fig. 5 that after the annealing, the dislocations in the (110) plane are disappeared completely, that is, it is just dislocations with anti-parallel Burgers vectors that existed therein at the preliminary straining. The second cause is the low shear stress value in the above-mentioned four easy slip planes. However, dislocations are formed and move therein at the bending under high loading. In the crossing sites with the (110) plane, the dislocation reactions of the (4) type occur resulting in formation of sessile dislocations.

The comparative consideration of kinetic creep curves under uniaxial compression and under bending of NaCl type single crystals oriented in various manners, the dislocation structures observed in experiment, and possible dislocation reactions, in our opinion, provides an important conclusion

concerning the mass formation of anneal-resistant ensembles of sessile dislocations. For the effective running of the dislocation reactions, not only the Frank energy criterion should be met, but certain dislocation kinetics conditions should take place. The dislocation reaction should have time to proceed over the whole (or over the majority) of crossing line of the slip planes of interacting dislocations. This means that the dislocations being involved in the reactions should have a low mobility. The mobility of the reacting dislocations is low both in the crystals compressed along the $\langle 111 \rangle$ direction and in the bent $\{110\}$ oriented crystals due to low reduced shear stresses. This results in mass running of the dislocation reactions causing the formation of sessile dislocations.

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Особливості високотемпературної анізотропії пластичної деформації монокристалів з ґраткою типу NaCl в умовах повзучості

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Досліджено особливості високотемпературної повзучості монокристалів NaCl та KCl при двох видах напруженого стану. Встановлено зв'язок поміж кількістю систем легкого ковзання та швидкістю деформування. Проаналізовано умови, за яких дислокаційні реакції у кристалах з ґраткою типу NaCl можуть мати місце, та результати взаємодії дислокацій.