

The stress-strain state of deformed NaCl type single crystals with the sample height smaller than the base side

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A real modeling of the mass transfer process in a crystal by visualization of the crystal material motion under deformation using markers as well as structure studies have revealed inhomogeneity in the stress fields and in the crystal dislocation structure. Four crystal regions have been distinguished differing in the dislocation structures and the dislocation interaction mechanisms. For each region, the structure evolution has been studied and the structure state diagram has been constructed as a function of the pre-straining rate.

Реальное моделирование процесса массопереноса в кристалле путем визуализации, с помощью маркеров, перемещения кристаллического материала при деформировании и структурные исследования выявили неоднородность полей напряжений и неоднородность дислокационной структуры кристаллов. Выделены четыре области кристалла, характеризующиеся различными дислокационными структурами и механизмами междислокационного взаимодействия. Для каждой из областей исследована эволюция структуры и построена диаграмма структурных состояний в зависимости от степени предварительной деформации.

It is known [1, 2] that, under plastic deformation of single crystals of NaCl lattice type oriented along [100] relatively to the loading and having the height (a) to base side (c) ratio about 2 to 3, the gliding in the initial deformation stages occurs along the {011}<011> planes tilted at 45° to the loading direction.

The plastic deformation of single crystals with $a/c < 1$ is specific in that the dislocation exit out of the gliding strips to the surface in the central region is blocked by the deforming setup rods. The studies on metals [3, 4] have evidenced that when the $a/c < 1$ samples are deformed by compression, a bulk stress-strained state (SSS) arises in the crystal due to the plastic strain inhomogeneity. In the works cited, polycrystalline samples were considered,

thus, the sample structure consisted of a set of mutually misoriented grains, and the dislocation exit out of the gliding strips to the surface was blocked by grain boundaries. There are no literature data on the SSS studies in NaCl type crystals being deformed under the loading under consideration. Those differ from the metals in that there are only three possible active gliding systems (<110>, <100>, and <111>) acting at 45° and 90° to each other. The plastic strain inhomogeneity might result in inhomogeneous stress relaxation for various crystal regions and thus in various dislocation structures (the dislocation interaction mechanisms). In this connection, of importance is to study the specific features of NaCl lattice type crystals deformed in the

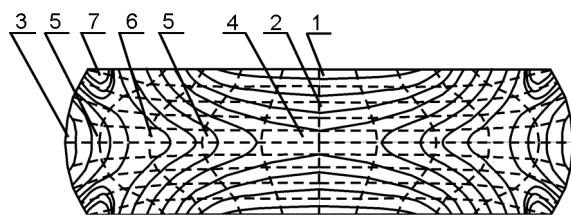


Fig. 1. Distribution of the material shift diagrams in the simulated crystal at ε values (per cent): 18 (1), 25 (2), 27 (3), 28 (4), 29 (5), 30 (6), 42 (7). Dotted line shows the FE matrix shift of the sample.

case when the sample height is smaller than the base side.

Simulation. To calculate SSS (in particular, to study the material shift distribution and stresses in the crystal), the COSMOS/M software was used developed at Structural Research and Analysis Corp., 1661 Lincoln Boulevard, Suite 100 Santa Monica, California 90404, 2000, where the SSS of an object is calculated using the finite element (FE) method. The simulation of the plastic straining process by the FE method had the following specific features: (i) when simulating the strain up to stresses corresponding to the plastic strain process onset (yield limit), the calculation accuracy using FE between the stress distribution and the material shift in a real (physical) and simulated (virtual) crystal depends on the initially preset physical and mechanical characteristics of the material; and (ii) when the crystal being simulated is strained up to stresses exceeding the yield limit, the FE calculation does not take into account the changes in the internal stress fields caused by the multiplication and motion of dislocations. Only the macro-scale changes in the internal stresses was accounted for by means of setting the plastic flow law for a group of FE. That factor was described as a strain-induced hardening curve obtained in experiment for a crystal being deformed in conditions where the dislocation interaction processes typical of a certain group of FE dominate therein.

Since it is just the fcc lattice that is typical of NaCl, KCl, and NaI crystals, it is just KCl that was selected as the model material to study the structure peculiarities under inhomogeneous SSS due to its low hygroscopicity. The sample to be simulated having the ratio $a/c < 1$ was coated by a rectangular FE grating where the mechanical characteristics of KCl single crystal were preset [5]. For the FE group arranged in the

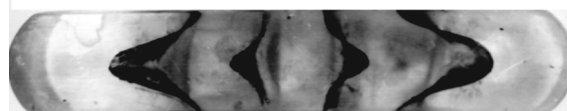


Fig. 2. Material mass transfer in a crystal under straining.

central region of the virtual crystal (where the dislocation exit to the surface in a real crystal is blocked by the deforming setup rods), a plastic flow law (the strain-induced hardening curve) was set obtained for the samples strained at $a/c < 1$. For the peripheral FE group arranged in the region where the dislocation exit to the surface is possible in a real crystal, the plastic flow law was set obtained for the samples with $a/c = 2.5$. Fig. 1 presents the cross-section of the crystal being simulated along its vertical symmetry axis at the relative strain of the sample as a whole equal to 25 %. The dotted line shows the FE shift in the matrix while the solid one, the "iso-stressed lines". Considering that picture, it is to note that at the moderate crystal straining near the upper and lower surfaces, the material relative strain increases from 18 % to 42 % when passing from the center to periphery.

Experimental. The material was deformed using an Instron testing machine. The sample dislocation structure was visualized by selective etching with PbCl_2 in ethanol.

To compare the material shift patterns in the physical and virtual crystals, a modeling experiment was carried out. The $50 \times 50 \times 10 \text{ mm}^3$ plates were cleaved out of a single crystal ingot along the cleavage planes. The dislocation density was 10^6 cm^{-2} . The surface was coated by 0.1–0.2 mm thick graphite layer. The plates were stacked together as a sandwich. The sample so obtained was heated up to a pre-melting temperature, strained by 2 to 5 % and hold under loading for 2 h. As a result, the sintering boundary was decorated by well visible black micro-particles. Then the sample was unloaded and turned by 90° . The repeated straining of the sample evidence a shift of decorating black points. In fact, the experiment provides a shift diagram of all the crystal points under straining at elevated temperatures. In Fig. 2, shown is the cross-section of a sample set up by 50 %. The material shift picture obtained under plastic flowing of a physical crystal is similar to that of the finite-elemental matrix in a virtual crystal, thus evidencing the admis-

sibility of the obtained calculated values of the material strain in the crystal.

Discussion. When considering the strain distribution diagrams, the horizontal (perpendicular to the compression axis) inhomogeneity as well as the vertical (along the compression axis) one are to be distinguished. As to the horizontal inhomogeneity, the material strain value increases from the center to the periphery and decreases at the crystal edge. As to the vertical inhomogeneity, the strain in the central region increases from the butts (contacting with the deforming setup rods) towards the center. At the periphery, an inverse process takes place, that is, the relative strain decreases from the butts to the center.

The inhomogeneous plastic flow of the material and the corresponding inhomogeneity in the internal stress distribution must result in an inhomogeneous stress relaxation due to dislocation multiplication and motion. As a consequence, two specific features are to be noted in the stress relaxation at the crystal straining. First of all, the dislocation structure studies have shown that at the periphery where the dislocation exit onto the side surface is possible, the structure differs heavily from that of regions positioned near the crystal center where the dislocation exit onto the surface is blocked by the deforming setup rods. The photo in Fig. 3 illustrates those crystal zones (right, the region where the dislocation exit onto the surface is blocked by the deforming setup rods; left, that where the dislocation exit onto the side surface is possible). The interface between those regions is in the vertical cleavage plane at 45° from the crystal edge to the horizontal symmetry axis. The somewhat unusual look of the dislocation structure photo is associated with the structure study difficulties in the selected conditions of the crystal deforming. So at the material strain of 3 to 5 %, the dislocations which cannot go to the crystal surface are accumulated therein. Therefore, at the chemical etching, only a high dislocation density is observed and the small-angle boundaries forming the substructure are difficult to distinguish. That is why it is difficult to estimate the internal substructure parameters. In this connection, the procedure was used providing the etching-off of single dislocation and clear visibility of the dislocation walls (boundaries) only. It is known [6] that the distance between the dislocation walls allows to judge the internal stresses in a crystal. A smaller inter-

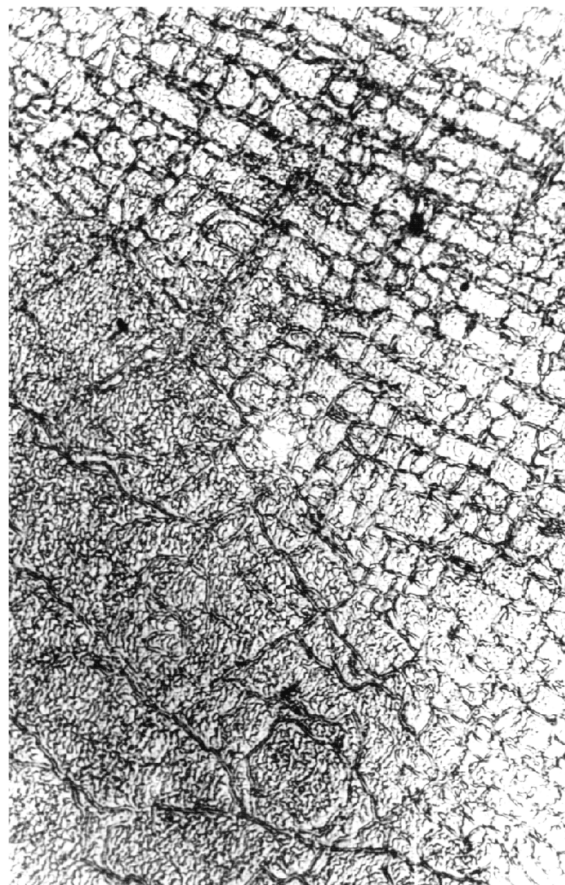


Fig. 3. Etching pattern of transversal cross-section of the crystal region where various dislocation interaction mechanisms are acting ($\times 70$).

wall distance is typical of higher stresses. Thus, the sharp difference in dislocation structure on the crystal area of $2 \times 2 \text{ mm}^2$ size (Fig. 3) evidences the difference in the internal stress levels and seems to be associated with different mechanisms of dislocation interaction at the crystal periphery and in its center. This confirms that the selected calculation model is valid when the plastic straining is simulated using the FE method.

Another specific feature of the stress relaxation consists in that at the initial plastic straining stage, when the dislocation structure can be studied by optical microscopy, the dislocation density near the contact surfaces at $\varepsilon = 0.6 \%$ increases from 2.5×10^6 to $6 \times 10^6 \text{ cm}^{-2}$ and the material relative strain increases by a factor of 2.3. This phenomenon can be interpreted as a result of hindered stress relaxation within the region where the material shift is small. Thus, the residual stresses at plastic straining decrease from the center to periphery.

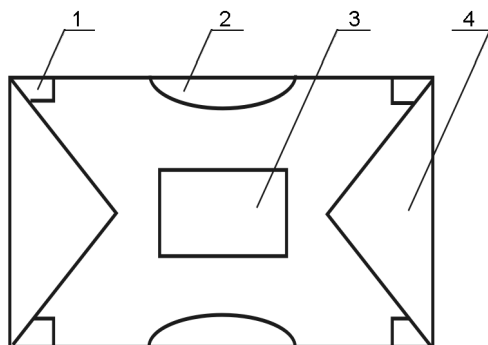


Fig. 4. Scheme of crystal regions with different stress relaxation intensities under plastic straining: the equal material shift region (1); the hindered straining one (2); the intense straining one (3); the region where dislocation exit to the crystal surface out of the gliding strips is possible (4).

The whole set of the results obtained allow to distinguish several characteristic regions in the crystal with specific dislocation structures inherent therein (see Fig. 4). First, there is a region of the equal material shift arranged near the horizontal symmetry axis. It is characterized by the material shifting inhomogeneity of maximum 8 %. The relative strain value is in correlation with ε value for the crystal as a whole. The second region is that of hindered straining. It is at the center near the contact surfaces. The material is subjected to volume compression. As a result, the material shift in that region is minimal and amounts 20 to 30 % less than that in the equal material shift region. Thus, the stress relaxation is also hindered in that region. The third region is the intense mass transfer one arranged at the periphery near the contact surfaces and characterized by the most intense material straining (low level of residual stresses). At last, there is a region of the possible dislocation exit to the surface. It is arranged at the periphery near the contact surfaces. In that region, the dislocation interaction mechanisms are typical of crystals strained at a/c ratio amounts 2 to 3.

The material plastic straining inhomogeneity and effects of various dislocation interaction mechanisms result in different structure states of different crystal regions, the strain value of the crystal as a whole being the same. Fig. 5 presents the structure state diagram for KCl, NaCl, NaI(Tl)

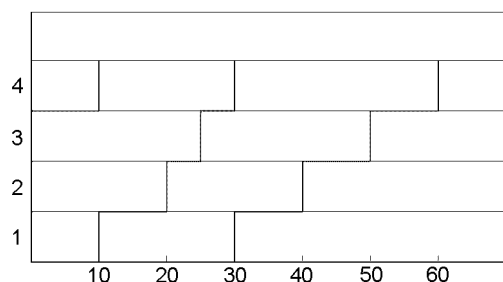


Fig. 5. Structure state diagram for crystals of NaI(Tl) type lattice strained at the height smaller than the base side: the intense straining region (1); the equal material shift one (2); the hindered straining one (3); the region where dislocation exit to the crystal surface out of the gliding strips is possible (4).

crystals strained at $T = (0.5 \text{ to } 0.75)T_m$ for different a/c values from 0.25 to 1.

To conclude, the strain-stressed state of crystals with $a/c < 1$ is strongly inhomogeneous and is due to two factors. The first one is the inhomogeneous material shift resulting from the fact that the tangential stresses generated by friction at the contact surfaces (of the samples and deforming elements) are distributed non-uniformly over the crystal height and amount to a maximum at the contact surfaces and to a minimum at the crystal half-height. Another factor is the edge effect associated with the possible exit of dislocations to the side surface. This results in an inhomogeneous distribution of strains and stresses as well as of the subsequent stress relaxation and of the accompanying residual stress pattern in the crystal. This is confirmed both by experimental studies of the crystal dislocation structure and by calculations of the stressed state.

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**Напружено-деформований стан кристалів
із ґраткою типу NaCl, деформованих в умовах,
коли висота зразка менше сторони основи**

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Реальне моделювання процесу масопереносу у кристалі шляхом візуалізації, за допомогою маркерів, переміщення кристалічного матеріалу під час деформування та структурні дослідження виявили неоднорідність полів напруг та неоднорідність дислокаційної структури кристалів. Виділено чотири області кристала, які характеризуються різними дислокаційними структурами та механізмами взаємодії. Для кожної з областей досліджено еволюцію структури та збудовано діаграму структурних станів залежно від ступеня попередньої деформації.