EVOLUTION OF THE DEFECT STRUCTURE IN ALKALI-HALIDE CRYSTALS AT THE PLASTIC DEFORMATION OF BIG-SIZE LARGE-APERTURE CO_2 LASER WINDOWS

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We present here a complex experimental study of the optical quality and the strength to laser radiation of alkali-halide crystals (AHC) thermomechanically strengthened using different working modes. We have revealed in this study the change of deformation mechanism, according to the schemes {110}→{110} *and* {100}<110>→{100}<110> *that occurs at an increased deformation degree, from* 5 *to* 60%, *at temperatures from* 200 to 400°C. *It is shown that formation of a coarse-grained re-crystallization structure at the deformation degree above* 25–40% *leads to an enhanced absorption at* 10.6 μm *wavelength and weakens the AHC strength to laser radiation.*

Low mechanical strength of alkali-halide crystals (AHC), the material that is most common in the high-power optics, makes it very urgent the task of improving their mechanical properties while keeping their optical quality.

It is known that the crystal strength is related to the dislocation displacement process, deceleration of which is feasible by forming impurity or intrinsic defects when doping a crystal, making a substitution solid solution or when strengthening a single crystal by deformations (Refs. 4–10).

The doping of AHC using inovalent dopants, for example Ca^{2+} and Ba^{2+}, improves their mechanical properties, but strongly deteriorates their optical quality, in particular, the absorption at 10.6 μm wavelength increases by more than one order of magnitude (Ref. 5). The substitution solid solution KCl–KBr doubles the tensile strength while decreases the heat conductivity of mixed crystals and makes the stoichiometric ratio variable over the ingot volume thus causing the instability of the crystal strength (Refs. 5 and 10).

Obtaining of a fine-grained polycrystal structure through uniaxial deformation and recrystallization of single crystals is the simplest and most technological way of crystal strenthening (Refs. 6, 8, 9, and 11). Plastic deformation creates high-density dislocations in the crystal slip planes, and the strengthening occurs due to difficulties for the displacements of secondary dislocations (Ref. 6).

When compressing a prism or cylinder-shaped specimen along its maximum dimension there appears a homogeneous stress in the specimen. The physical processes taking place in thus deformed specimens are well studied (Refs. 5–9). The general deformation mechanism in this case is the slip of dislocation through the easiest slip systems. In the crystals of NaCl type the plastic deformation due to compression along the direction <001> results, at room temperature, in multiplication and displacement of dislocations along the directions <001> and <011> (Ref. 12). The system of easiest slip is {110}<110>, that makes the specimen to take a barrel shape.

When deforming a plate-shaped specimen the crystal takes an inhomogeneous stressed state. In this case it is of special interest to investigate the deformation regularities for technologies of making high-power infrared optics that possesses high mechanical strength.

Optical components with the size from 60×60×8 to 175×175×21 mm have been made from the deformation-strengthened pieces of KCl and NaCl. The deformation-worked pieces have been ground without any abrasive material until a required size is reached. The final polishing of the working facets has been performed using diamond powder. The strengthening of AHC is performed on a deformation machine of a stringent type at temperatures from 200 to 400°C by compressing the single-crystal plates along the direction <001>. Investigation of the defect structure evolution has been carried out using a polarization technique and analysis of the material microstructure. The influence of the strengthening regime on the AHC optical quality is estimated from the dependence of the absorption coefficient at 10.6 μm wavelength and of the breakdown threshold W_{th} on the degree of crystal deformation ε.

To study the laser radiation strength of the deformation-strengthened AHC, we used a large-aperture TEA CO_2 laser delivering 1μs-duration, at half height level, pulses. The spot of laser radiation of 25–240 cm^2 area in the plane of the specimen is shaped using a 100-element raster-mirror optical system. The mean radiation energy density varied in the range from 0.5 to 30 J/cm^2.

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The patterns, observed in polarized light, of the deformation-strengthened plates (Fig. 1) and their comparison with the data of microstructure analysis (Table I) shows a nonuniform distribution of the residual stresses and reveals the structure reconstruction that occurs in the plate during its deformation strengthening. The main cause leading to this situation is high complexity of the deformation process in a big-size plate due to inhomogeneity of the stress at a compression of a rectangular-shaped ingot. In this case different possibilities exist for the active deformation in the different zones of the plate. It is known that even in the case of homogeneous stress the plastic deformation never occurs in the entire volume of the crystal deformed. Nonuniform distribution of the local internal stress may occur in a crystal before its deformation. As a result, the shear can occur randomly thus yielding a nonuniform distribution of the shear during the plastic deformation. Different macroscopic (crystal surface, inclusions, and pores) and microscopic (dislocations and point defects) defects may cause the appearance of the local stress in the crystal.

One of the causes of the stress inhomogeneity is skin friction, that means that the displacements of the particles of AVH plate and the velocity of this displacement relative to the press are different at different points of the contact area. Thus, in the central part of the plate the skin friction should be lower than on its edges. Besides, the observed pattern of the residual stresses is the result of at least two process, that is of the plastic deformation and of the residual stresses relaxation. The processes of the stress relaxation in the different zones of the plate are also different.

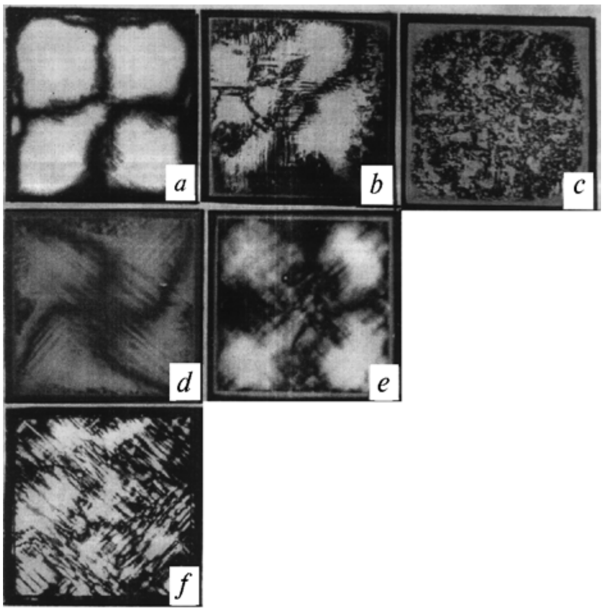

FIG. 1. Typical birefringence patterns after deformation of AHC at the temperature of 400°C *to the deformation degree* 10% *(a),* 40% *(b),* 60% *(c); at* 300°C *to* 10% *(d),* 40% *(e),* 60% *(f).*

TABLE I. Characteristic size (in μm*) of grains and type of the recrystallization structure.*

ε, %	Temperature, °q , at which the deformation is performed			
	200	300	350	400
5–10	2–6 micro-grain, polygonization	3–8 micro-grain	3–15 micro-grain with grain up to 100	30–200 early stage of collecting recrystallization
15–20	3–10 micro-grain, polygonization	10–40 micro-grain with grain up to 100	10–200 early stage of collecting recrystallization	200–3000 large-grain, collecting recrystallization
25–40	–	20–1000 early stage of collecting recrystallization	10–5000 large-grain, collecting recrystallization	20–5000 large-grain, collecting recrystallization
50–80	–	–	100–1000 large-grain, collecting recrystallization	500–10000 large-grain, collecting recrystallization

The pictures of the typical patterns of the birefringence of crystals KCl, with the deformation degree ε = 10–60% and temperature 200–400°C (see Fig. 1) show that birefringence pattern changes in to depend on temperature and degree of deformation in the following way.

In AHC, deformed at $t < 200$°C to $\varepsilon < 10\%$ (Fig. 1*f*), birefringence bands are observed only along the <110> direction. With the increase in temperature and degree of deformation to 200°C and 15–20% and 300°C and 10% (Fig. 1*d*) the clear zones are observed along with the bands along the direction <110>, i.e. zones of stress appear at the centers of the deformed plates. The bands along <110> are observed here against the background from bright spots, but already along the mutually perpendicular directions like <110>

and in other zones of the plate. In this case a homogeneous large-grain structure forms with the characteristic grain size of 2–10 μm.

When deformed at t = 300°C to ε = 20–40% and at t = 350–400°C to ε = 5–10% the specimen exhibits the birefringence bands directed along the directions like <100> together with the <110> bands (Fig. 1*a*,*e*). At increasing temperatures and deformation degree the number of the latter bands also increases. Thus, in the crystals deformed at t = 350–400°C to ε = 20–40% only the bands along the <100> direction and stressed zones in the centers of plate quadrants are observed (Fig. 1*b*).

The deformation of AHC at 300°C to 15–20% and at 350–400°C to 10% leads to the formation of a coarse-grain structure with individual large grains of 100–150 μm size. In the crystals deformed at 300°C to 25%, at 350°C to 15–20%, and at 400°C to 10% the early stage of collecting recrystallization, starting in the edges of the ingot deformed is observed. With the increase in temperature and deformation degree the processes of the dynamic and collecting recrystallization in the crystals deformed at t > 350–400°C to ε > 25–30% become more intense. As a result a coarse-grain recrystallization structure is being formed with the grain size from hundreds of microns to tens of millimeters, as it is seen in the polarized light in the form of spots of stress (Fig. 1*c*).

As known (see, e.g., Ref. 8) in the AHC at high temperatures in the presence of internal stress and when no deformation along the <110> is already possible the deformation according to the slip system {100}<110> can occur. This explains the occurrences of the birefringence bands along the <100> direction to be the result of the stress increase due to the shear displacement along the <100> direction. In this situation carrying out of the material along the direction <100> leads to rounding off of the rectangular ingot at increasing temperature and the deformation degree. This is confirmed by the measurements of deformation degree along the direction where the material is carried out at different values of t and ε along the directions of a load action presented in Table II. The sequence of the birefringence pattern change also is an evidence of the deformation mechanism change for the slip in the planes {100} with the temperature increase up to 300–400°C and the deformation degree above 20–25%.

The investigation results showing the influence of the deformation-strengthening regimes on the AHC strength to laser radiation and on its optical quality are shown in Fig. 2.

As is seen from this figure the absorption coefficient at 10.6 μm wavelength sharply increases with ε increasing above 40%. Simultaneously there occurs a decrease of the laser breakdown threshold, so that with the increase of ε to 50–60% and higher the laser breakdown threshold inside the volume becomes even lower than the surface breakdown threshold.

TABLE II. Values of the deformation degree along the directions of material carrying out, <110> *and* <100>, *for different regimes of strengthening.*

t, °C	ε <001>, %	ε <110>, %	ε <100>, %
20	7	1.5	2
200	10	3	4
300	37	17	33
300	30	15	28
350	50	28	47
400	20	11	22
400	39	20	42
400	57	25	56

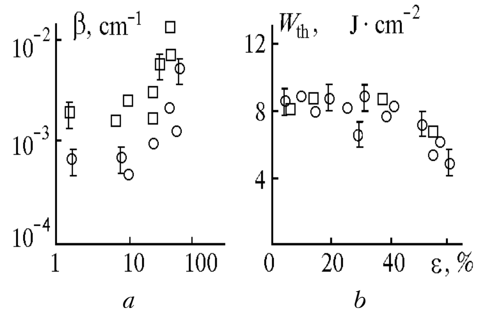

FIG. 2. The absorption coefficient (a), and the energy density threshold of spatial damages occurrence (b) as functions of the deformation degree in NaCl (□) *and* KCl (o) *at temperatures from* 300 *to* 350°C.

This regularity can be explained by an increase in the concentration of absorbing micro-impurities on the boundaries of blocks and grains. The area of the latter ones decreases approximately by 10^5–10^8 times with the change of the small-grain structure for a large-grain one, that is accompanied by carrying out of the micro-impurities from the grain volume to its boundary.

Typical stress-strain diagrams of KCl crystals, that have been strengthened under different regimes are shown in Fig. 3.

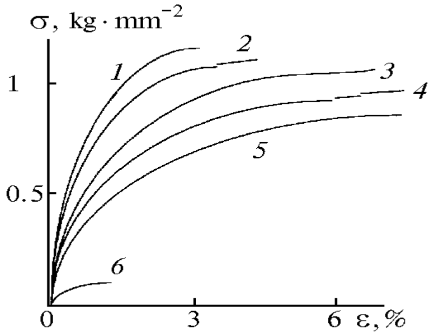

FIG. 3. Typical stress-strain diagrams of KCl *crystals, that have been strengthened under different regimes: at* 200°C *to* 20% *(1), at* 300°C *to* 15% *(2), at* 400°C *to* 15% *(3), at* 400°C *to* 37% *(4), at* 400°C *to* 60% *(5), and that of a single crystal (6).*

From the dependences depicted it follows that thermomechanical working of the AHC allows one to increase the fracture strength, σ, by 5 to 7 times. The crystals strengthened at $t = 300–400°C$ are more plastic, while the deformation in crystals strengthened at $t < 200°C$ exhibits higher brittleness of the AHC. Therefore the ingots for making optical element should preferably be worked at higher temperatures, since in this case the risk for the ingot to be cleft at a deformation decreases. At the same time certain loss of strength is observed in the specimens worked at $t > 350–400°C$ and $\varepsilon > 40\%$ as compared to those strengthened at $t < 300°C$ and $\varepsilon < 15–25\%$.

The comparison of the results on laser breakdown threshold and absorption coefficient of the strengthened AHCs made with the results of microstructure and polarization analysis, and strength investigation shows that the unstable large-grain structure of collecting recrystallization formed deteriorates their optical quality and strength to laser radiation. In contrast, the deformation strengthening of AHC at temperature 300–400°C to the deformation degree 30–40% does not practically influence on the crystal optical quality. The most uniform structure is obtained by deformation of AHC along a single axis at temperature below 300°C to the degree of 10–25%.

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