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Thermoelastic stresses and their relaxation at alkali-halide single crystals hardening

O.I.Kovtun[2], D.V.Matsokin[1], I.N.Pakhomova[1]

[1]V. Karazin Kharkov National University, 4 Svobody sq., 61022, Kharkiv, Ukraine
matsokin@univer.kharkov.ua
[2]Physikalisches Institut der Universität Heidelberg, Im Neuenheimer Feld 226, 69120 Heidelberg, Germany

The origin of internal stresses during hardening of alkali-halide single crystals was investigated. It is shown that the thremoelastic stress relaxation is accompanied by the fragmentation of distinct areas of a single crystal and by the development of new dislocations, which formed a cellular structure. The internal stress distribution is qualitatively analyzed by the photoelasticity method.

Keywords: hardening; thermoelastic stress; dislocation; fragmentation; photoelasticity.

Экспериментально исследовано возникновение внутренних напряжений при закалке щелочногалоидных монокристаллов с решеткой типа NaCl. Показано, что релаксация термоупругих напряжений сопровождается возникновением новых дислокаций, формирующих ячеистые структуры; и фрагментацией отдельных областей монокристалла. Методом фотоупругости качественно проанализировано распределение внутренних напряжений.

Ключевые слова: закалка; термоупругие напряжения; дислокации; фрагментация; фотоупругость.

Експериментально досліджено виникнення внутрішніх напружень при загартуванні лужногалоїдних монокристалів з граткою типа NaCl. Показано, що релаксація термопружних напружень супроводжується виникненням нових дислокацій, які утворюють комірчасті структури, та фрагментацією окремих частин монокристала. Методом фотопружності якісно проаналізовано розподіл внутрішніх напружень.

Ключові слова: гартування; термопружні напруження; дислокації; фрагментація; фотопружність.

Introduction

Thermoelastic stresses (TES) always develop during the growth of dielectric crystals, in particularly alkali-halide. Thermoelastic stress relaxation can significantly change the crystal structure and affect further operating characteristics.

Some experimental observations of structural changes while hardening of alkali-halide single crystals with NaCl-type lattice are presented in this article. The majority of the performed experiments used KBr single crystals, as such crystals are relatively "soft" and do not crack during hardening

Experimental technique

Experiments were performed with alkali halide single crystals of 10x10x10 mm size with initial dislocation density $\rho \sim 10^{5}\ \text{cm}^{-2}$. Crystals were heated on a ceramic substrate at constant rate $W = 6\,\frac{\text{K}}{\text{min}}$ to temperature *T*, then maintained at this temperature for a particular time *t* and quickly taken out from the oven (to the room temperature). For the KBr single crystals: $T = 620°\text{C}$, $t = 15\ \text{min}$.

The cooled down crystals were cleaved, the dislocation structure along with the cleavage relief were optically analyzed. By the photoelasticity method the distribution of the internal stresses was studied [1].

Thermoelastic stresses

As well known, cooling begins from the surface. Hence the near-surface layer tends to shrink and thus compresses the internal volume. The situation is similar to the stretching of a metal hoop on a barrel. As a result, in the internal region the compressing stresses appear, while stretching stresses, parallel to the closest crystal side, develop in the near-surface layer. The mean value of these stress can be estimated from a ratio:

$$\sigma \approx \varepsilon E \approx \alpha \Delta T E \qquad (1)$$

where $\varepsilon = \alpha \Delta T$ is the relative crystal strain in the near-surface layer parallel to it's surface, α is the linear thermal expansion coefficient, $\Delta T = T - T_r$ is the difference between the temperature of the heated crystal and the room temperature T_r, *E* is Young's modulus in the direction

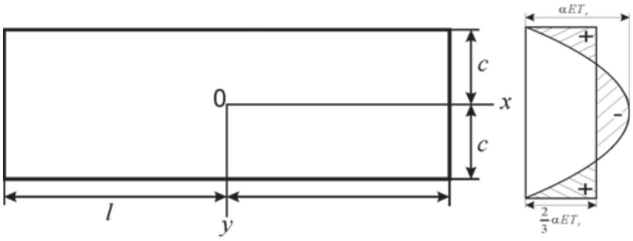


Fig. 1. Thin rectangular plate of constant width 2*c*. Temperature is an even function of *y* and does not depend on *x* and *z*.

parallel to the crystal surface. In our case $\Delta T \approx 600\ \mathrm{K}$ and $\alpha \approx 3\cdot 10^{-5}\ \mathrm{K}^{-1}$ [2], so after removing of the crystal from the oven the internal stresses reach $\sigma \approx 1.8\cdot 10^{-2}\ \mathrm{E}$. This value considerably exceeded the level of stress necessary for dislocation arising in alkali halide crystals.

The TES problem is not solved analytically for isotropic cubic shape bodies [3, 4]. There are solutions for several simple cases of temperature distribution and for rotationally symmetric figures (sphere and cylinder). In particular, for a thin rectangular plate of constant width 2*c* (Fig 1a) in which the temperature is an even function of *y* and does not depend on *x*, namely

$$T = T_r\left(1 - \frac{y^2}{c^2}\right)$$

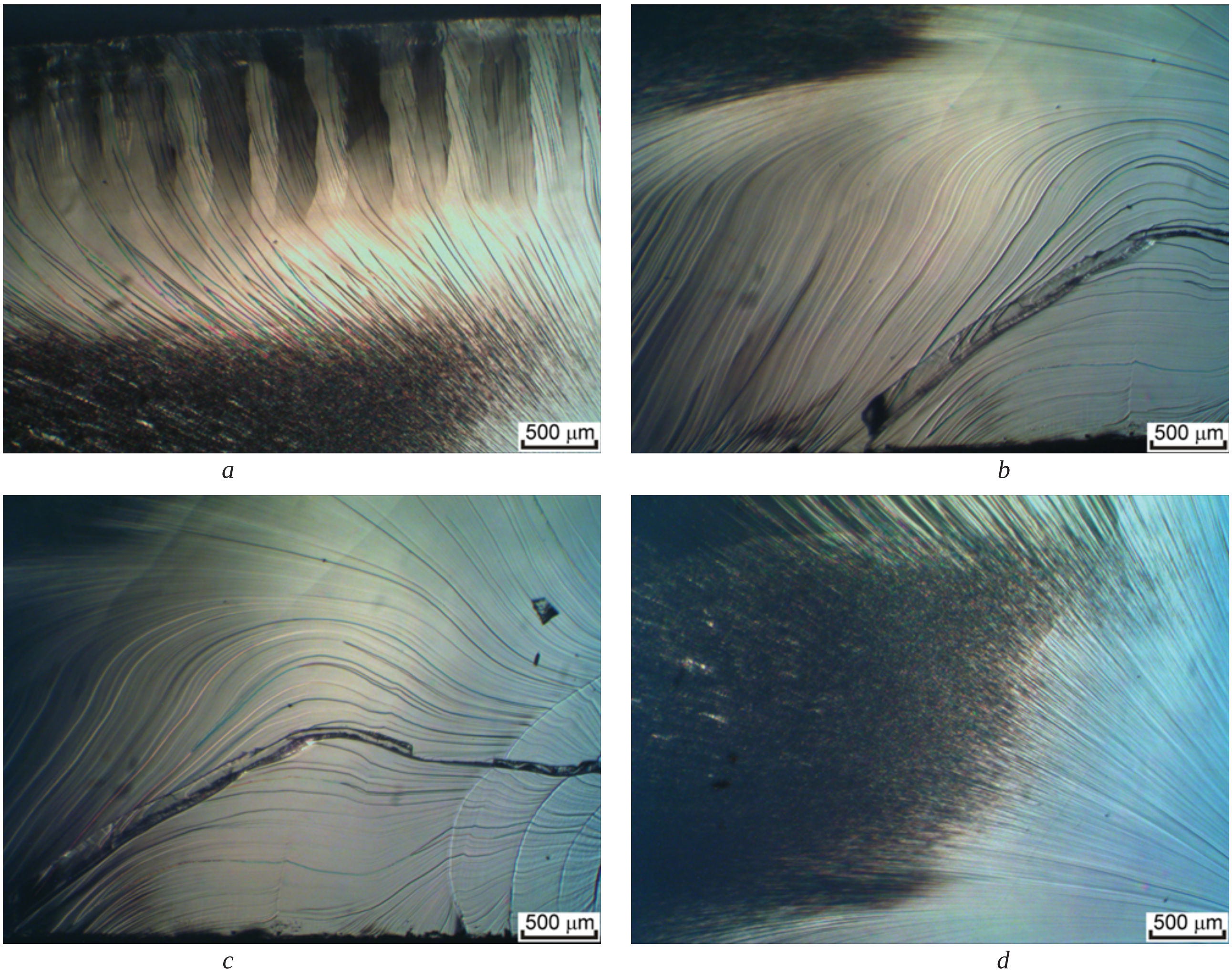
The solution for TES is [3]

$$\sigma_x = \frac{2}{3}\alpha T_r E - \alpha T_r E\left(1 - \frac{y^2}{c^2}\right) \qquad (2)$$

(see Fig. 1b)

If a sphere of temperature T_0 is dipped into a liquid with temperature T_1 ($T_1 > T_0$) the external part of the sphere will be dilate thus causing the comprehensive uniform radial stretching in the middle. The maximum value of this stress is

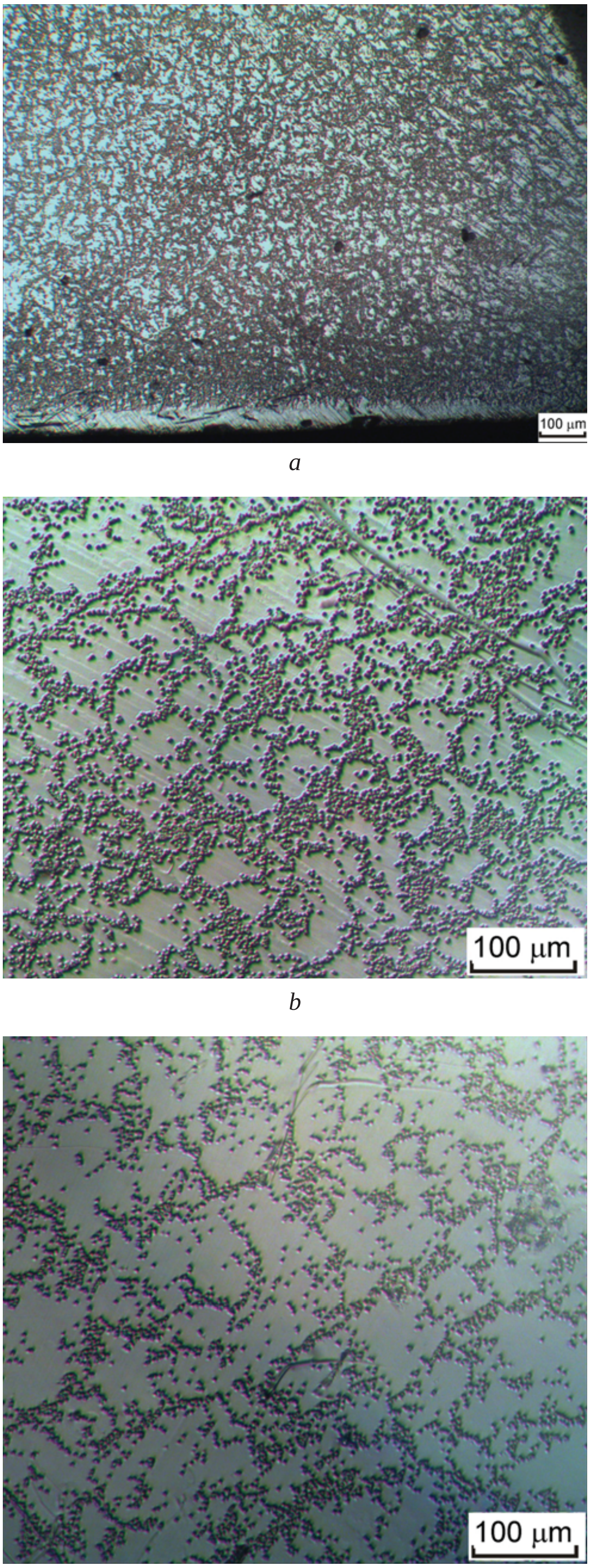
$$\sigma_r = 0.771\frac{\alpha E}{2(1-\nu)}(T_1 - T_0) \qquad (3)$$



a *b*

c *d*

Fig. 2. The cleavage of hardened KBr single crystal. $T = 620°\mathrm{C}$, $t = 15\ \mathrm{min}$.



a

b

c

Fig. 3. Dislocation structure of a hardened sample: a – near to the surface, b, c – in the middle.

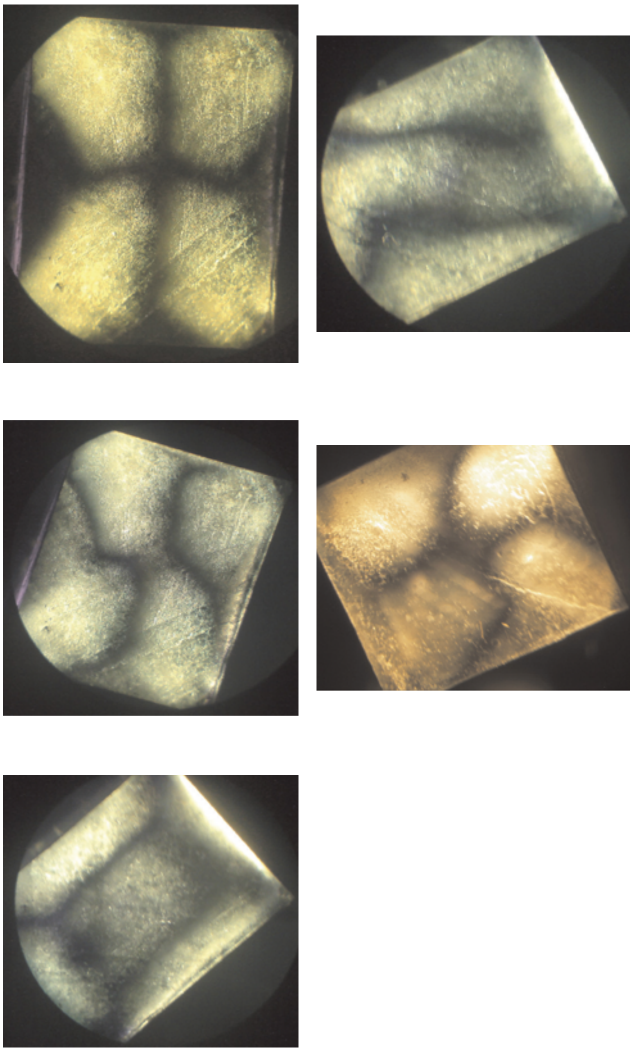


Fig. 4. Photoelastic pictures of KBr single crystals. Isocline parameters: a – 0°, b – 25°, c – 30°, d – 40°, e – 45°.

at time

$$t = 0.0574 \frac{R^2 C\rho}{\chi} \qquad (4)$$

In (3) and (4) ν is Poisson’s ratio; *R* – sphere radius; C, ρ, χ – heat capacity, density and heat conductivity.

Results

Surface relief.

1) Periodically located bands of reorientation near to a surface of hardened single crystals.

2) Dense system of cleavage steps; in the outer layer the step orientation becomes normal to the surface (Fig. 2 b, c).

3) The cleavage step density is the greatest in the central part of crystal.

Dislocation structures.

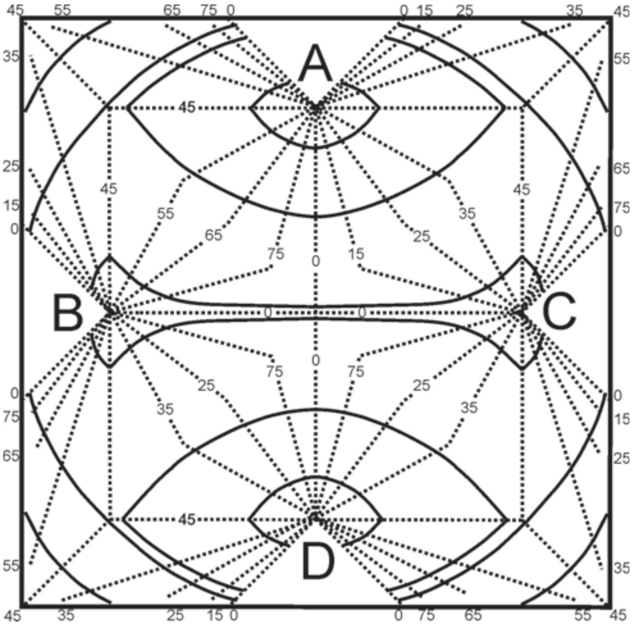


Fig. 5. Image of isoclines (dotted lines) and isostates (solid lines) in a hardened crystal.

The resulting dislocation density is greatest near to the surface. In the central part of the crystal, dislocations form a cellular structure with diffused boundaries (Fig. 3b, c).

The dislocation density is significantly decreased after annealing of the hardened single crystal at $T = 625°\mathrm{C}$ for $t = 3\ \mathrm{h}$. This effect can be explained by the annihilation of dislocations with antiparallel Burger's vectors. Cleavage steps became curved and their density decreased.

Photoelastisity.

Observations of the hardened single crystals in polarized light (light source⇒polarizer⇒crystal ⇒analyzer) in the crossed nicols yielded interesting photoelastic images that changed as the crystal was rotated with respect to the light polarization plane (Fig. 4).

Two principal stresses σ_1, σ_2 and the shear stress τ_{12} are presented in each point of plain stressed body. The relation between stresses

$$\tan 2\vartheta_{1,2} = \frac{2\tau_{12}}{\sigma_1 - \sigma_2}.$$

Black lines in Fig. 4 are isoclines along which the angles (ϑ_1, ϑ_2) between principal stresses σ_1, σ_2 and polarization plane [1] are preserved.

The crystal was rotated with respect to the light polarization plane from 0° to 90° in steps of 5°. A schematic isocline pattern is shown in Fig. 5. Each isocline is marked with its parameter. As seen from the figure there are four points of intersection of the isoclines of different parameters (A, B, C, D). So-called "special isotropic points" are where only the hydrostatic pressure is present. Solid lines in Fig. 5 denote isostates; at each point the tangent is consistent with the direction of one of the main stresses.

The difference between principal stresses $\Delta\sigma = \sigma_1 - \sigma_2$ using isochromatic (color) lines could not be determined, as the optomechanical coefficient proportional to $\Delta\sigma$ is small in alkali-halide crystals [1].

The obtained pictures qualitatively obviously show the presence of internal stresses in hardened crystal.

Conclusions

The internal thermoelastic stresses emerge in alkali halide single crystals during hardening. Relaxation of these stresses leads to significant change of the crystal structure up to the transformation of some regions into polycrystal by reorientation and fragmentation.

References

1. Frocht, M.M., Photoelasticity. J. Wiley and Sons, London, 1965.
2. P. D. Pathak, J. M. Trivedi, N. G. Vasavada, Thermal expansion of NaF, KBr and RbBr and temperature variation of the frequency spectrum of NaF, Acta Cryst. (1973). A29, 477-479.
3. Sadd M.H. Elasticity. Theory, Applications, and Numerics. Elsevier. 2005, 473 p.
4. Boley B. A., Weiner J. H. Theory of Thermal Stresses, 1960 (John Wiley, London).