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# Thermal activation analysis of the dislocation unpinning from stoppers in KCl crystals

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The effect of temperature and elastic static loading on the dislocations unpinning from stoppers in KCl crystals has been studied. Dependence of the stress of the activation energy on activation volume of the studied process has been determined, value of the energy of the dislocation binding with the pinning center has been calculated. Based on the comparison of the power law of interaction of dislocations and the stopper with the existing theoretical laws, there has been made a conclusion about the possible nature of the dislocation blocking by pinning centers in these crystals.

**Keywords**: elastic deformation, activation energy, activation volume, binding energy.

Вивчено вплив температури та пружного статичного навантаження на процес відкріплення дислокацій від стопорів у кристалах KCl. Визначено залежності від напруження енергії активації та активаційного об'єму досліджуваного процесу, розраховано величину енергії зв'язку дислокації з центром закріплення. На основі зіставлення одержаного силового закону взаємодії дислокації та стопору з існуючими теоретичними законами зроблено висновок щодо можливого характеру блокування дислокацій центрами закріплення у досліджуваних кристалах.

**Ключьові слова**: пружна деформація, енергія активації, активаційний об'єм, енергія зв'язку.

Изучено влияние температуры и упругого статического нагружения на процесс открепления дислокаций от стопоров в кристаллах KCl. Определены зависимости от напряжения энергии активации и активационного объема изучаемого процесса, рассчитана величина энергии связи дислокации с центром закрепления. На основании сопоставления полученного силового закона взаимодействия дислокации и стопора с существующими теоретическими законами сделан вывод о возможном характере блокирования дислокаций центрами закрепления в исследованных кристаллах.

**Ключевые слова**: упругая деформация, энергия активации, активационный объем, энергия связи.

## Introduction

The study [1] offered a theory for conducting thermal activation analysis of the dislocation unpinning from pinning centers basing on results of acoustic measurements. These measurements were obtained under simultaneous influence of temperature and stresses applied to samples in the elastic strain range. To test this theory [1], authors studied ultrasonic attenuation $\alpha_d$ in high-purity copper samples on a frequency of 10,17 MHz under the influence of thermomechanical effects mentioned above. As a result, temperature dependence of average length of dislocation segment $\ell$ (T) was determined as well as an average size of dislocation cell L and the binding energy of impurity atom with dislocation. Having analyzed obtained data and chemical composition of studied samples, authors [1] have made a conclusion that atoms Fe and Zn are the most probable centers of dislocation pinning. Note that previously thermal activation analysis was conducted basing on data obtained by measuring individual dislocation mobility [2,3] or by applying low-frequency internal friction KHz frequency range [4,5]. But obtained data were always unreliable. Method [2,3] errors occur due to difficulties in calculation of stress that secure passage of individual dislocation on ~ 50 interatomic spacing in crystal when individual dislocation is under alternate influence of direct and reverse mechanical impulse. Errors in the second method [4,5] occur because changes in dislocation structure of crystal caused by influence of elastic waves of considerable amplitude are not taken into consideration. All this leads to distortion of observed thermal activation processes [6]. The following method [1] is deprived of these limitations. According to this method sounding pulse with deformation amplitude in sound wave $\varepsilon \sim 10^{-7}$ is passed through the crystal under the external thermomechanical effects. The acoustic signal allows to get high accuracy information on subtle processes in crystals at their loading in the field of quasi-elastic deformation, and thus does not affect the course of their occurrence. Due to certain technical difficulties associated with its implementation, the method [1] has not received wide application, although the relevance of its application to the study of the power laws of interaction of dislocations with stoppers [7], of course, remains relevant.

Until recently, there were only two works, performed on CsJ [8] and KBr [9] single crystals, in which the thermal activation analysis was conducted in the framework of the theory [1]. Taking into account the recommendations provided in the study [1], authors [8-9] have received a similar array of experimental data but applied theoretical patterns of power laws for their analysis [3]. As a result, the process of identifying the type of pinning centers in KBr and CsJ crystals has become more simple and informative. More recently, results of a new study [10] were published. The paper studied the temperature dependence of the average length of a dislocation segment $\ell(T)$ and the concentration of the stoppers $C(T)$, as well as the binding energy of a dislocation with a stopper in KCl single crystals was evaluated.

The purpose of this study is to continue similar investigations on the KCI single crystals, initiated in paper [10]. These investigations are aimed to determine the dependence of activation energy $U(\sigma)$ and activation volume $\gamma(\sigma)$ from stress and further comparison with theoretical curves [3], calculated for the most probable for our crystals dislocation pinning centers.

### Methodology of the experiment

In this study, as in [10], the dislocation ultrasonic $\alpha_d$ absorption in KCI single crystals was studied as a function of the statistic loading value of σ in 300-430 K temperature range. The value of $\alpha_d$ was measured by the superimposed exponent method on 7,5 MHz longitudinal wave. KCl crystal was chosen as the object of the study due to its low Debye temperature (Θ = 235K), which made it possible to observe the effects of the breakout of dislocations at a sufficiently low temperature tests. The test samples of $10^{-4}$ wt % purity and size of 17x17x23 mm$^3$ were obtained by chipping them along the cleavage plane <100>. The samples obtained were treated by grinding and polishing to achieve nonparallelism of the work surfaces of about ±1 mkm/cm. Then the prepared samples were annealed in the muffle furnace MP-2UM for ~12 h at temperature of 0,8 $T_{melt}$ ($T_{melt}$ = 770°C) with subsequent slow cooling up to the room temperature. High-temperature liquid VKJ-94 was applied as a transition layer between the piezoelectric transducer and the sample. Experiments were carried out by the following scheme. Using a specially designed furnace with electronic control a given test temperature of the samples was set and strictly maintained. The temperature was measured with a differential copper-constantan thermocouple. After reaching the required temperature an initial attenuation $\alpha_0$ at $\sigma = 0$ was measured. Then the sample was step-by-step loaded by compression in the tensile-testing machine of «Instron» type at strain rate of $\sim 10^{-5} s^{-1}$ in the range of stresses $\sigma = (0 \div 7) \times 10^5$ Pa. To avoid the point defect redistribution under applied load action the time of each loading was ~ 15 s. After each stop of the machine rod the quantities $\alpha_d$ and σ were measured. The sample was unloaded when the indicated loading range was over. It was found that after the sample unloading the quantity $\alpha_d$ returned to its initial value at the given temperature that evidence on the elastic character of the sample loading. This has been confirmed by absence of strain stress on the chart of the recorder KSP-4 at each stop of machine movable clamps in the indicated range of temperatures and stresses. The corresponding estimates were carried out to exclude apparent losses as well as losses caused by the nonparallelism of the working surfaces of the sample from the measured ultrasound attenuation [11]. Analysis has shown that the dominant contribution in the apparent loss is made by the diffraction loss $\alpha_{theor}$= 0,15 Db/mks and the contribution from other above-mentioned losses is negligibly low.

### Results and discussion

Experimental dependences $\alpha_d$ (σ), obtained at temperatures 300, 340, 385 and 430 K are presented in Fig. 1. The curves for temperature range 300 – 400 K are presented in paper [10]. As it is shown in Fig. 1, the importance of stress value applied to samples σ at increase in acoustic losses $\alpha_d$ becomes more considerable when the temperature of experiment increases. According to the authors [10], this is explained by lower level of potential barrier for dislocations that try to unpin from pinning centers when crystals are heated. We have used theory [1] to make preliminary analysis of the distraction unpinning from stoppers under simultaneous influence of temperature and elastic static loading. The theory is based on the

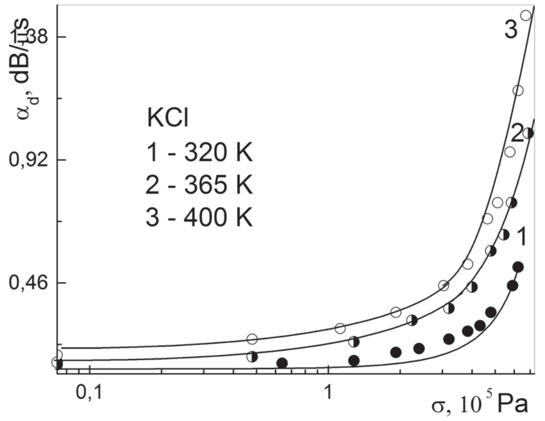

*Fig.1.* Dislocation ultrasonic absorption $\alpha_d$ in KCI as a function of the elastic statistic loading at different temperatures.

expression [7] for probability $W$ of dislocation segment unpinning from one of point stoppers of total number $N = L/\ell$ under external static loading $W(\sigma,T)\approx N\cdot\exp(-\frac{U(\sigma,T)}{kT})$. Here $U(\sigma, T) = U_0 - \Upsilon\tau$ is energy of activation of the dislocation unpinning from the pinning centers, $U_0$ is energy of the dislocation binding with the pinning center in the absence of external stresses; $\tau = \Omega\cdot\sigma$ is the reduced shear stress provoking the dislocation slipping; $\Omega$ is orientation factor taking into account that the reduced shift stress in the slip plane is less than the applied stress; $\Upsilon = b\cdot d\cdot\ell$ is the activation volume, where $d \sim (1\div3)\,b$ is distance of the effective dislocation binding with the stopper, $b$ is the Burger vector. In the approximation of the catastrophic dislocations unpinning from stoppers authors [1] have accepted the relation that takes into account the change of the dislocation segment length when the external stress changes in the form of: $\ell(\sigma, T) = LW + \ell(1 - W)$.

Taking into account that $\alpha \sim l^4$ [7] the relation is used for low frequencies $\left(\frac{\alpha_d(\sigma)}{\alpha_0}\right)^{\frac{1}{4}} \approx \frac{\ell(\sigma)}{\ell}$, where $\alpha_0 = \alpha_d(\sigma = 0)$. The resulting relation, appropriate for thermal activated analysis based on experiment data, looks like [1]:

$$\ln\left[\left(\frac{\alpha_d}{\alpha_0}\right)^{\frac{1}{4}} - 1\right] \approx \ln\frac{L^2}{\ell^2} - \frac{U_0}{kT} + \frac{\Omega b d\ell\sigma}{kT}. \qquad (1)$$

Using dependences $\ln\left[\left(\frac{\alpha_d}{\alpha_0}\right)^{\frac{1}{4}} - 1\right] - \sigma$ for different T within 300 – 430 K range, authors [10] have determined the temperature dependence of average length of dislocation segment $\ell(T)$ and stoppers concentration $C(T)$. The diagram $C(T)$ allows to determine energy of the dislocation binding with stoppers $U_0$. In accordance to [1]:

$$\ln C \approx \ln\left[\left(\frac{\beta_a}{\beta_m}\right)\cdot C_0\right] - \frac{(S_m - S_a)}{k} + \frac{U_0}{kT}, \qquad (2)$$

where $\beta_a$ and $\beta_m$ are numbers of equivalent free positions of interstitials or substitutions in the unit cell of the dislocation atmosphere, $C_0$ — atomic fraction of impurity atoms in the matrix dimension, $S_a$ and $S_m$ — vibrational entropies of matrix atoms and dislocation atmosphere. It is seen that having the dependence $\ln C - f(1/T)$ it is easy to determine $U_0$ by the slope. In the paper [10], we have found value $U_0 \sim 0{,}35$ eV for KCl crystals that is in good agreement with the results of [1,8-9]. In addition, the average size of dislocation cell was determined $L = 4{,}2\times10^{-6}$ m, that is in good accord with the data of [12]. This paper generalizes the experimental data presented in [1] and those listed in Figure 1 After processing the entire array of data for KCl in the framework of relation (1) we can determine the dependence on the activation energy of the voltage $U(\sigma)$ and the activation volume $\gamma(\sigma)$. For this purpose a series of cross-sections was made $\ln\left[\left(\frac{\alpha_d}{\alpha_0}\right)^{\frac{1}{4}} - 1\right] - \sigma$ for all the studied temperatures in 300 - 430 K range for different values of loading. The stress value at which the section of curve was made $\ln\left[\left(\frac{\alpha_d}{\alpha_0}\right)^{\frac{1}{4}} - 1\right] - \sigma$ for each of the temperatures, was selected from the linear portion of the given dependence where the condition of the catastrophic dislocations unpinning from stoppers was fulfilled [1]. The higher the temperature of the experiment was, the smaller the critical stress value $\sigma^*$ was needed for passage to the linear area. The results are shown in Fig. 2. It can be seen that for all stresses in the $0{,}5 - 6\times10^5$ Pa all the experimental points fit well a straight line from the slope of which the function $U(\sigma)$ can be easily obtained. Using the values of $\Omega = 0{,}42$, $b = 4{,}46\times10^{-10}$ m, and data $\ell(T)$ [10], the activation energy as a function of the stress $U(\sigma)$, shown in Fig. 3 as curve 1, was obtained. The curve extrapolation at zero stress of the curve gives energy of the dislocation binding with the stoppers. Continuing dotted experimental curve $U(\sigma)$ to $\sigma = 0$, we obtain $U_0 = 0.35$ eV, which agrees well with the value $U_0$, calculated from the slope of the

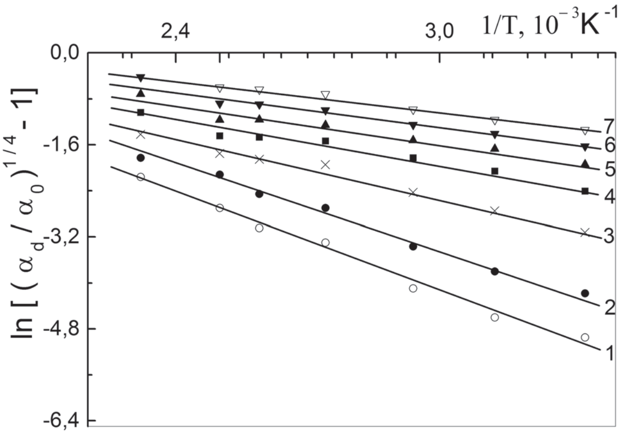

*Fig.2.* The logarithm of the dislocation ultrasonic absorption $\alpha_d$ in KCl as a function of converse temperature at different values of elastic static loading σ, Pa : 1 – $0{,}5\times10^5$; 2 – $1\times10^5$; 3 – $2\times10^5$; 4 – $3\times10^5$; 5 – $4\times10^5$; 6 – $5\times10^5$; 7 – $6\times10^5$.

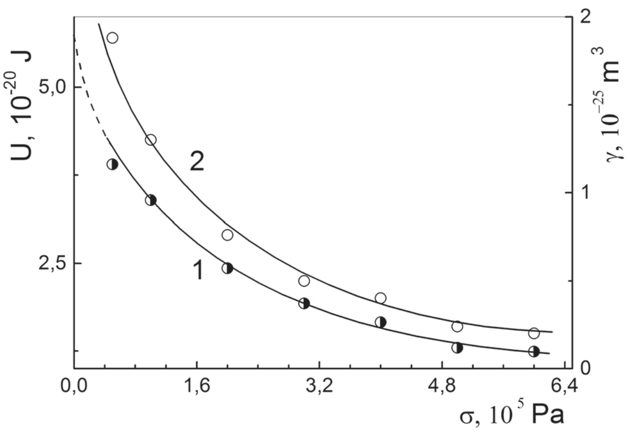

*Fig.3.* The activation energy (1) and activation volume (2) in KCI crystals as a function of the value elastic static loading.

curve $\ln C$ - $f(1/T)$ [10]. Further, by graphical differentiation of the curve $U$ ($\sigma$) we obtain the dependence of the activation volume of the voltage for the dislocations unpinning from stoppers (Figure 3, curve 2). It can be seen that both $U$ and $\gamma$ characteristics decrease with increase of $\sigma$, this indicates a decrease in the level of the potential barrier for dislocation under such conditions and explains the behavior of the temperature curves 1-3 shown in Figure 1. Indeed, with an increase in stress the value of the activation energy of thermal dislocations unpinning from stoppers reduces, so the long dislocation loops appear, and by law $\alpha \sim l^4$ ultrasonic absorption increases that is observed in the experiment. To compare the data obtained with known from literature power laws of interaction of dislocations with pinning centers we reconstruct dependence $\gamma(\sigma)$ in the reduced coordinates ln ($\sigma/\sigma^*$) – ln ($\gamma/\gamma^*$). As $\sigma^*$ was taken the maximum stress of studied interval $7\times10^5$ Pa and the corresponding value $\gamma^*$ on the curve $\gamma(\sigma)$. Fig. 4 shows a comparison of experimental data with the theoretical profiles 1 and 2 [3] (where 1 - interaction [211] of edge dislocations with <100> tetragonal defects, and 2 - interaction [101] of screw dislocations with <110> tetragonal defects). It can be seen that both interaction mechanisms are effective and it is likely that dislocation in the investigated KCl crystals interact with both types of stoppers.

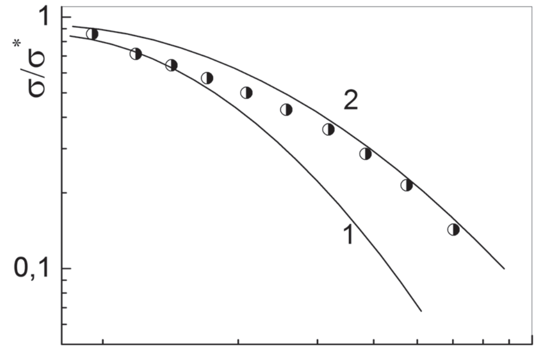

*Fig.4.* The change in the reduced activation volume depending on the reduced stress. Points - the results of the present study, 1 and 2 - power laws [3] of interaction [211] of edge dislocations with <100> tetragonal defects and [101] screw dislocations with <110> tetragonal defects respectively.

## Conclusion

1. The acoustic impulse method was applied to investigate, in the temperature range from 300 to 430 K at 7,5 MHz frequency, the dislocation absorption $\alpha_d$ ($\sigma$) in KCI crystals as a function of the elastic statistic loading.

2. The dependence of the activation energy and activation volume of the external static loading have been determined. It has been found that when stress increases both mentioned characteristics decrease, this means that the value of energy barrier for thermal dislocation unpinning from stoppers decreases. The data obtained explain the behavior of curves $\alpha_d$ ($\sigma$) at a fixed value of the loading under conditions of temperature increase. The amount of energy $U_0$ of the dislocation binding with the stoppers was calculated 0,35 eV. This result is in good accord with the value $U_0$ [10] found in a different way.

3. A comparison of the power law of the dislocations binding with stoppers, obtained by experiment and theoretical profiles [3] calculated for the case of interaction [211] of edge dislocations with <100> tetragonal defects and [101] screw dislocations with <110> tetragonal defects. It is shown that both interaction mechanisms are effective and it is likely that the dislocations in the investigated KCl crystals interact with both types of stoppers.

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