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THE INVESTIGATION OF X-RAYS IRRADIATION EFFECT ON THE MOBILITY OF DISLOCATIONS IN LiF CRYSTALS

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An acoustic pulse echo method at a frequency of 7.5 MHz was used to study the dependence of the absorption α and the relative velocity of ultrasound $\Delta v / v_0$ in single crystals of LiF with a residual deformation of ε = 0.65%, at 300 K in the irradiation dose range 0...1057 R. It was found that an increase in the irradiation dose leads to a noticeable decrease in the damping and the relative ultrasound velocity in the sample as a result of a decrease in the effective density of the dislocation segment *L* when it is fixed by radiation centers. Based on the results of measurements of acoustic characteristics, the absolute value of the damping factor of dislocations *B* is determined and compared with the literature data obtained from the high-frequency branch of dislocation resonance.

INTRODUCTION

One can see from the reviews [1–4] that a lot of theoretical and experimental works have been devoted to the study of the radiation exposure effect on the properties of crystals. It became known that an increase in the number of radiation centers in ionic crystals stimulated the appearance of acoustic effects caused by the interaction of these point defects with mobile dislocations. To mark out the deposition process onset at dislocations of different pinning points, the low-frequency methods of amplitude-dependent internal friction are proved to be the most effective [1, 2]. With their help, the smallest changes in the average effective length of the dislocation loop *L* oscillating in the field of the ultrasonic wave can be clearly recorded. These changes, by virtue of the law $\alpha \sim L^4$ [3] have a very significant effect on the measured attenuation α magnitude of ultrasound in the sample. The noted methods are very sensitive to the appearance of radiation centers in the crystal, although their application is fraught with certain difficulties. Firstly, when a low-frequency wave of high amplitude is transmitted, deformation by the ultrasound may occur in the crystal under investigation, which can lead to a change in both the value of *L* and the dislocation density Λ. In addition, at large amplitudes of the ultrasonic wave, small portions of radiation defects can not exhibit an appreciable stopping effect on a mobile dislocation. Therefore, low-frequency experiments often use large doses of radiation, which can lead to changes in mechanical [1, 5–7] and optical [8–17] crystal characteristics.

In this connection, to solve the mentioned problems, we began to use the amplitude-independent, acoustic method of the MHz-frequency range [18, 19], which is highly informative and highly sensitive to the effect of weak radiation stoppers on the dislocation. Analyzing a large array of experimental data [18–26, 28, 29] on the dynamics of dislocations under conditions of various external influences, it is not difficult to see that only those results obtained on the descending branch of a damped dislocation resonance are correct. The experimental data obtained at the low-frequency branch of this resonance are preliminary in nature and therefore need serious verification and refinement. Due to this, the purpose of the present paper is to determine the value of the coefficient of dynamic braking of dislocations B from low-frequency measurements and a comparison with the analogous value found from the high-frequency asymptote of dislocation resonance.

MATERIALS AND EXPERIMENTAL TECHNIQUES

In this paper, we studied the effect of weak X-ray irradiation on the absorption and ultrasound velocity in LiF single crystals with a residual deformation of 0.65% at a frequency of 7.5 MHz at a temperature of 300 K. The samples having parameters 17×17×27 mm and purity of 10^{-4} weight %, obtained by puncturing on cleavage planes <100>, were used in the experiments. The attenuation of ultrasound was measured with the method of superimposing a calibrated exponent on the reflected echoes, and the impulse interference method as well as the selector method [2], realized by the apparatus [27], were used to measure the velocity. In order to carry out precise acoustic measurements, the studied samples, in accordance with recommendations [20–26, 28, 29], were subjected to fine optical polishing after puncturing, so that the non-parallelism of their working faces was ± 1 μm/cm, which was monitored with an IKV type optimeter. An additional estimate of the non-parallelism degree in the “piezo-quartz-glue-sample” system could also be obtained in the process of superimposing a calibrated exponent on a series of reflected pulses observed on an oscilloscope screen. To remove internal stresses that could arise during machining of samples, they were annealed for ~ 12 hours in MP-2UM muffle furnace at a temperature of ~ $0.8 \cdot T_m$ (where T_m is the melting temperature), followed by slow cooling to room temperature. In order to implant “easy-moving” (mobile) dislocations into the crystal, it was preliminarily deformed to obtain a residual deformation of ε = 0.65%. The deformation of the samples was carried out by compression in a rupturing machine of the Instron type at a speed of ~ 10^{-5} s^{-1}. Under this deformation regime, slip bands do not arise, and etch pits uniformly cover the etched surface of the crystal [20–26, 28, 29], which allows us to determine the dislocation density in crystals quite accurately. The technology for the dislocation component's isolation from the total absorption was the same as in [20–26, 28, 29].

The irradiation of the investigated LiF crystals was performed at a standard X-ray unit of the URS-55 type, which provided the radiation dose rate ~ 0.11 R/s at the location of the investigated crystal. To avoid the appearance of possible inhomogeneities in the sample caused by internal mechanical stresses [1, 4], each of the 4 lateral faces parallel to the long axis of the crystal was irradiated alternately for 20 min, which corresponded to a total dose of 1057 R.

RESULTS AND DISCUSSION

Fig. 1 shows the results of studying the dislocation absorption's dependence α on the time of X-rays irradiation for LiF samples with a residual deformation of ε = 0.65%. It is evident that with an increase in the irradiation time of the sample – the attenuation of ultrasound decreases sharply. It should be noted that even after the first 20 min of irradiation, the attenuation decreased by 0.34 dB/μs, and during the remaining measurement time its value decreased by another 0.3 dB/μs. Completely expected were the results, shown in Fig. 2, obtained on the same sample when measuring the relative velocity of ultrasound $\Delta v/v_0$, where $\Delta v = v_0 - v$ (v_0 – speed in the initial annealed sample, measured at a frequency of 217.5 MHz).

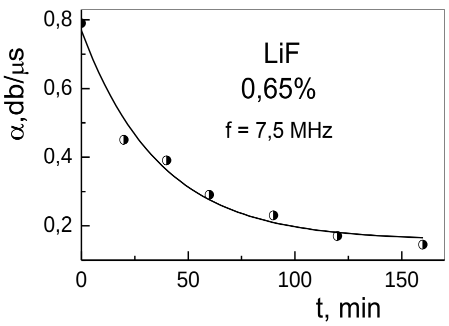

Fig. 1. The dependence of the dislocation ultrasound absorption on the irradiation time in LiF crystals with a residual deformation of 0.65% at T = 300 K

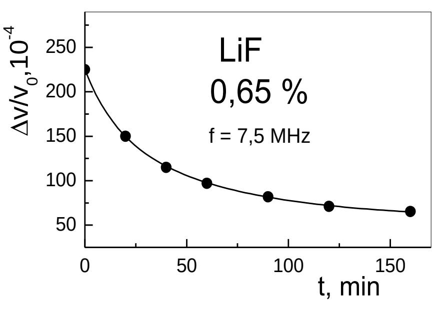

Fig. 2. The dependence of the relative propagation velocity of ultrasonic waves on the irradiation time in deformed (0.65%) LiF crystals at T = 300 K

One can see that as the irradiation time increases, the effect of decreasing the relative propagation velocity of the ultrasonic wave in the crystal is observed. We couldn't help note, that an analogous behavior of the acoustic characteristics in the course of the irradiation time increasing was also found earlier on NaCl crystals [18]. Using the data on $\Delta v/v_0$ and α, in paper [18] the calculation of the dislocation structure's parameters Λ and *L* was carried out by means of formulas for low frequencies [3]:

$$\Lambda = 8.68\,10^{-6}\left(\frac{\pi^4 B}{\Omega G b^2}\right) f^2 \left[\frac{(\Delta v/v_0)^2}{\alpha}\right], \quad (1)$$

$$L = \frac{1}{2v}\left[\frac{10^6}{8.68}\frac{C}{B}\frac{\alpha}{\Delta v/v_0}\right]^{1/2}. \quad (2)$$

The values of the dislocation characteristics Λ and *L* were determined in [18] as a result of calculations, and they were found to be $1.2\cdot10^{11}$ m^{-2} and $1.3\cdot10^{-6}$ m, respectively, and it was assumed that $B = 10^{-5}$ Pa·s.

To clarify the correctness of the results presented in [18] and for the comprehensive verification of the theory [3], we suppose, that it is important to calculate the coefficient of viscosity *B* from the formula (1) and compare it with the estimation of *B* found in [24, 29] on the high-frequency asymptote of a damped dislocation resonance. The expression for the value *B*, using formula (1), takes the form:

$$B = \frac{\Omega G\, b^2 \Lambda_e}{8.68\,10^{-6}\pi^4 f^2}\frac{\alpha}{(\Delta v/v_0)^2}, \quad (3)$$

where Λ_e is the density of dislocations found by the etch pit method; *Ω* is the orientation factor; *C* is the linear tension of the dislocation; *G* is the shear modulus; *b* is the value of the Burgers vector; *f* is the operating frequency of the ultrasonic wave.

With the help of the substituting into the formula (3) the values *Ω* = 0.311 [24, 29]; $G = 3.533\cdot10^{10}$ Pa [24, 29]; $b = 2.85\cdot10^{-10}$ m; $f = 7.5\cdot10^6$ Hz; $\Lambda_e = 1.74\cdot10^{10}$ m^{-2} [24, 29], as well as the data on $\Delta v/v_0$ and α shown in Figs. 1, 2, there were formed the dependence of the damping constant *B* on the irradiation time, presented in Fig. 3.

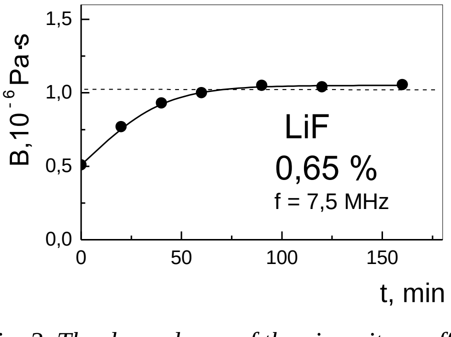

Fig. 3. The dependence of the viscosity coefficient B on the irradiation time in LiF crystals with a residual deformation of 0.65% at T = 300 K

It is obviously that the value of *B* is irrespective of the fact that the irradiation dose, and a small deviation in the first ~ 30 min of exposure can be a consequence of the fact that the initial deformation of 0.65% lowers the value of the resonant frequency. Because of this, according to [2, 3], the initial condition $\omega/\omega_m \ll 1$ may not be fulfilled strictly enough to simplify the derivation of relations (1) and (2). Measuring on the same LiF crystals the frequency dependences of the dislocation resonance in [24, 29], we've got the opportunity to found that before the irradiation, the resonance maximum was at the frequency of 14 MHz, and after 20 and 40 min of irradiation it moved to positions 25 and 40 MHz respectively. At the same time, the working frequency of 7.5 MHz, at which the values of α and $\Delta v/v_0$ were measured in this paper, already appeared on the ascending branch's linear part of the frequency profile [3], where the condition $\omega/\omega_m \ll 1$ is satisfied.

Due to the comparing the absolute value of $B = 1.05\cdot10^{-6}$ Pa·s, obtained in this paper with the estimate $B = 3.7\cdot10^{-5}$ Pa·s, found out from the high-frequency branch of dislocation resonance [24, 29] by using the "reference" value Λ_e, determined by the method of selective etching, was revealed the fact that these estimates of *B* are more than an order of magnitude different. Such a difference, according to [30], is caused by the impossibility of describing acoustic measurements conducted over a wide range of frequencies by using a single damping mechanism in the framework of the dislocation theory [3]. It is rather useful to take the obtained results into experiments, where the influence of various factors [31–43] on the mobility of dislocations in crystals is studied.

CONLUSIONS

There were investigated the effect of x-ray irradiation in the dose range 0...1057 R on the dislocation absorption $\Delta v/v_0$ and the relative ultrasound velocity in LiF single crystals, deformed to 0.65% and measured at a frequency of 7.5 MHz at room temperature. It was established by experiments that, after the first $t = 20$ min term of irradiation, the attenuation and relative change in ultrasound velocity in a crystal are substantially reduced. An attentive analysis of the obtained results showed that changes in these acoustic characteristics, closely connected with increasing radiation dose, are associated with a decrease in dislocation mobility due to their stopping by point radiation defects. On the ground of the obtained experimental data and the formula for low-frequency measurements of the Granato-Lucke theory, the absolute value of the damping factor of dislocations *B* and its behavior under the conditions of changing the irradiation dose of the sample was determined in this paper. The comparison of the value *B* with the analogous data found in special literature on the high-frequency branch of a damped dislocation resonance was successfully performed. It was vividly shown that these estimates of *B* found on different branches of the frequency profile of dislocation resonance differ by more than an order of magnitude.

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ИССЛЕДОВАНИЕ ВЛИЯНИЯ РЕНТГЕНОВСКОГО ОБЛУЧЕНИЯ НА ПОДВИЖНОСТЬ ДИСЛОКАЦИЙ В КРИСТАЛЛАХ LiF

А.М. Петченко, Г.А. Петченко, С.Н. Бойко

Акустическим импульсным эхо-методом на частоте 7,5 МГц исследованы зависимости поглощения α и относительной скорости ультразвука $\Delta v/v_0$ в монокристаллах LiF с остаточной деформацией ε = 0,65% при 300 К в интервале доз облучения 0…1057 Р. Обнаружено, что увеличение дозы облучения приводит к заметному снижению затухания и относительной скорости ультразвука в образце вследствие уменьшения

эффективной длины дислокационного сегмента L при его закреплении радиационными центрами. На основе результатов измерений акустических характеристик определено абсолютное значение величины коэффициента демпфирования дислокаций B и проведено его сравнение с литературными данными, полученными по высокочастотной ветви дислокационного резонанса.

ДОСЛІДЖЕННЯ ВПЛИВУ РЕНТГЕНІВСЬКОГО ОПРОМІНЕННЯ НА РУХЛИВІСТЬ ДИСЛОКАЦІЙ В КРИСТАЛАХ LiF

О.М. Петченко, Г.О. Петченко, С.М. Бойко

Акустичним імпульсним ехо-методом на частоті 7,5 МГц досліджено залежності поглинання α і відносної швидкості ультразвуку $\Delta v / v_0$ у монокристалах LiF із залишковою деформацією ε = 0,65% при 300 К в інтервалі доз опромінення 0…1057 Р. Виявлено, що збільшення дози опромінення призводить до помітного зниження згасання і відносної швидкості ультразвуку в зразку через зменшення ефективної довжини дислокаційного сегмента L при його закріпленні радіаційними центрами. На основі результатів вимірювань акустичних характеристик визначено абсолютне значення величини коефіцієнта демпфування дислокацій B і проведено його порівняння з літературними даними, одержаними за високочастотною гілкою дислокаційного резонансу.