ACOUSTIC STUDIES OF THE EFFECT OF X-RAY IRRADIATION ON THE DYNAMIC DRAG OF DISLOCATIONS IN LIF CRYSTALS

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The dislocation resonance in LiF single crystals with the residual deformation $\varepsilon = 1.5\%$ is studied by the pulsed method in the range of radiation doses 0–660 R and the frequency range 22.5–232.5 MHz at room temperature. Based on the analysis of the obtained results, it is established that the X-ray irradiation of the crystals results in a significant change of the frequency and amplitude localizations of the dislocation resonance due to the variation of the mean effective length of a dislocation segment, whereas the viscosity coefficient B remains constant.

1. Introduction

Investigation of the effects related to a variation of physical characteristics of crystals due to their irradiation is one of the priority problems in the physics of real crystals. It is known [1] that the effect of radiation on alkali halide crystals consists in the appearance of a large number of point defects in irradiated samples that can multiply (at high radiation energies) and unite into defects of complex configurations. Radiation damages change optical characteristics of solid bodies (resulting in the formation of color centers [1] or deformation-induced luminescence [2]), as well as their electrical characteristics [3–5], and induce variations of the density and the dimensions of irradiated samples due to the accumulated energy [1]. It is also known that a long-term irradiation can result in the crystal fracture [1]. Finally, it is exactly established that the irradiation has an effect on mechanical properties of solid bodies such as elastic moduli, hardness, and yield and ultimate strengths [1, 4–6]. Work [7] dealt with studying the effect of irradiation on the dislocation migration under the action of a magnetic field and proposed a new effective technique for revealing the effect of stoppers of various kinds on the dislocation drag.

A separate field in studying the effect of irradiation on physical properties of solid bodies is the investigation of the internal friction of irradiated crystals. Acoustic methods are very sensitive to any slight changes in the dislocation structure of a crystal arising in samples under irradiation. These peculiarities usually manifest themselves in the low-frequency region due to the fact that the ultrasound absorption at such frequencies is proportional to L^4, where L is the mean effective length of the dislocation segment that becomes a very sensitive experimental instrument [6, 8].

Reviews [1, 6] report on a number of experimental results obtained by various investigators and concerning with the effect of irradiation and, particularly, X-rays on the peculiarities of the ultrasound absorption in crystals at varying the temperature and the dislocation density. On their basis, an important conclusion was made that the hardening of alkali halide crystals is not due to F-centers themselves but due to accompanying interstitial halide atoms or ions or due to more complex ensembles of these defects. The radiation-induced defects play a role of dislocation pinning centers in the same way as impurity atoms [1].

It is worth noting that the majority of experimental results on the internal friction of irradiated crystals was obtained at a certain frequency in the amplitude-dependent region [1, 6], i.e. in the region of significant ultrasound loads, at which one registers an increase of the deformation of crystals due to the depinning of dislocations from Mott stoppers – nodes of the dislocation network, as well as the actuation of the Frank–Read source [6, 8].

It is clear that, at large amplitudes of ultrasound waves, oscillating dislocations practically do not feel the counteraction of "weak" stoppers (impurities, radiation damages, *etc.*), that is why the effect of X-ray irradiation can become noticeable at significant irradiation doses.

As concerns the effect of interaction between dislocations with stoppers on the initial irradiation stages, they can be fixed and thoroughly investigated using the precision technique of high-frequency amplitude-independent internal friction [8]. Applying this technique to researches of the behavior of the damped dislocation resonance, it is possible to correctly determine the absolute value of the constant of dynamic dislocation drag B that includes the total effect of all dragging forces acting on a dislocation and to reliably determine the effective length of a dislocation segment L that, to a large extent, depends on the presence of point defects in the crystal (including those of radiation origin).

It is worth noting that the indicated technique [8] was so far used mainly for studying the mechanisms of dislocation drag by phonons, electrons, and other dislocations. It was applied to researches of similar effects for irradiated crystals only episodically [6, 9, 10], particularly, when studying radiation damages in Cu crystals [6] under γ-irradiation or investigating the effect of X-rays on the dispersion of the high-frequency ultrasound velocity in NaCl [9]. In addition, this technique was used in [10] to study the effect of X-ray irradiation on the frequency spectra of acoustic losses for LiF crystals after their irradiation by a unit dose of approximately 10^3 R at $T = 300$ K. Based on the analysis of the obtained data, the authors of work [10] found out that X-ray irradiation results in a significant increase of the absolute value of the damping coefficient B from the initial value of 1.7×10^{-4} Pa·s (for an unirradiated sample) to 2.5×10^{-4} Pa· s after its irradiation. Unfortunately, neither the authors of work [10] nor other investigators succeeded in clarifying the reason for the indicated variation of the parameter B. That is why it is important to perform additional researches of this problem.

The aim of this work was to study in detail the effect of X-ray irradiation on the parameters of the damped dislocation resonance and to use them as a basis for obtaining the dynamic viscosity constant B and the effective length of the dislocation segment L as functions of the irradiation time t for LiF crystals. The obtained results can be useful both in the scientific and applied aspects, particularly, to expand the existing ideas about a fundamental characteristic of crystals (the dynamic viscosity coefficient B) and to adjust service characteristics of ion crystals used in various acousto-optical systems.

The investigation objects used in this work were LiF single-crystal samples. The behavior of the parameters B and L at the variation of the dislocation density Λ and the temperature T, as well as the effect of the change of these parameters on the localization of the frequency spectra of the dislocation ultrasound absorption $\Delta_d(f)$ for these crystals were studied in [11, 12].

2. Experimental Technique

The behavior of the frequency spectra of the dislocation decrement of the ultrasound absorption $\Delta_d(f)$ was investigated by the pulsed technique in the megahertz frequency range 22.5–232.5 MHz for LiF single crystals in the unirradiated and irradiated states. The magnitude of a preliminary deformation of the crystals necessary to introduce fast dislocations was equal to 1.5%. All investigations were performed at the constant temperature $T = 300$ K. The studied crystals represented $\langle 100\rangle$ samples $16\times16\times25$ mm^3 in size. To raise the accuracy of the acoustic experiment, the samples were ground and polished so that the nonparallelism of the working facets did not exceed $\pm1\mu$m/cm. The accuracy of a mechanical treatment was controlled by an IKV optimeter. The purity of the used crystals amounted to 10^{-4} wt%. To remove internal strains arising due to a mechanical treatment, the studied samples were annealed in a MP-2UM muffler during $\sim$12 h at a temperature close to the melting temperature of the crystal $T \sim 0.8T_m$ and after that slowly cooled down to room temperature. The preliminary deformation of the samples was performed using an "Instron" machine at a deformation rate of $\sim 10^{-5}$ s^{-1} by squeezing them along the crystallographic direction $\langle 100\rangle$. The choice of this deformation rate is nonrandom, as it was established [11–15] that, under such conditions, there are no slip bands, whereas the etching figures uniformly cover the etched surface. On the one hand, this simplifies the estimation of the dislocation density and, on the other hand, raises the accuracy of this calculation. To identify microphototographs more reliably, the calculations were performed with the use of the modern computer software Photoshop CS3 that allows one to automatically number and simultaneously to sum up etch pits, which eliminates random errors due to their repeated account. The samples were deformed with the help of an original device that made impossible distortions of the working facets of the sample when squeezing the crystals. Additional information concerning the etching, the method used to extract the dislocation component from the general absorption in the sample, and the technique of theoretical description of experimental points is given in more details in [11–15].

The X-ray irradiation of the crystals was performed on a standard URS-55 set-up with a copper anticathode and the working parameters $U = 40$ kV, $I = 10$ mA. The dose rate at the location of the crystals amounted to 0.11 R/s according to the data of a KID-2 dosimeter. In order to prevent the accumulation of inhomogeneous internal strains in the crystal [1], each of the three sides parallel to the long axis of the crystal was irradiated during 20 minutes except for the last (fourth) side that was irradiated during 40 minutes to determine the limit shift of the dislocation resonance to the high-frequency region. The total irradiation dose was equal to 660 R.

3. Experimental Results and Their Discussion

Figure 1 presents the experimentally obtained frequency dependences of the dislocation decrement $\Delta_d(f)$ for LiF crystals with the preliminary deformation $\varepsilon = 1.5\%$. According to [12], such a magnitude of the preliminary deformation for LiF crystals corresponds to a shift of the damped dislocation resonance toward higher frequencies due to the interaction of mobile dislocations with "forest" ones. It is interesting to find out if the used range of working frequencies is sufficient to register the shift of the $\Delta_d(f)$ curves to still larger frequencies due to the expected additional pinning [6] of dislocations by radiation defects. As one can see from Fig. 1, the X-ray irradiation of the samples considerably influences the characteristic shift of the resonance $\Delta_d(f)$ curves that monotonously move to the high-frequency region, by decreasing in height. It is easy to notice that, with increase in the frequency, the absorption changes with the irradiation time more slowly. The most pronounced effect of the quenching of the ultrasound absorption is observed for frequencies much lower than the maximum one due to the action of the locking law L^4 [6].

Analyzing the obtained data, one can see that the experimental curves $\Delta_d(f)$ have a form of the damped dislocation resonance [8], whereas the experimental points are well described by the theoretical frequency profile obtained in [16] for the case of the exponential length distribution of dislocation segments. According to the recommendations given in [8, 16], the experimental points were described by the theoretical profile mainly with regard for the points located at the decaying branches of the dependences $\Delta_d(f)$ (curves *1–4* in Fig. 1) and in the resonance region (curve *5* in Fig. 1). One can see that, at the maximal irradiation dose equal to 660 R

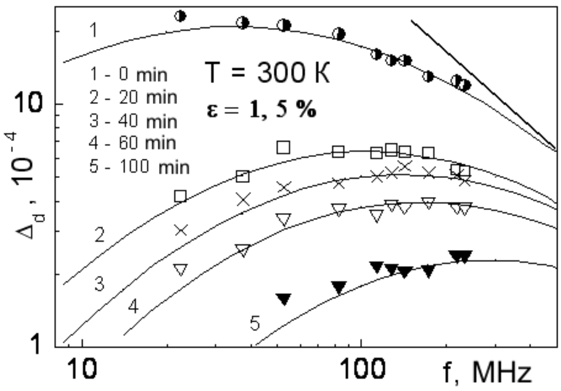

Fig. 1. Frequency dependence of the dislocation resonance absorption of ultrasound in LiF crystals preliminarily deformed to $\varepsilon = 1.5\%$ at $T = 300$ K at various irradiation times t, min: *1* – 0; *2* – 20; *3* – 40; *4* – 60; *5* – 100

(after 100 min of irradiation), the dislocation resonance shifted to the high-frequency region so far that it became impossible to use the after-resonance part of the experimental curve $\Delta_d(f)$. In other words, one can make an intermediate conclusion that, within the available interval of working frequencies of the measuring equipment, the limit irradiation dose for LiF samples with the residual deformation $\varepsilon = 1.5$ % at $T = 300$ K is equal to 660 R. In contrast to the frequency dependences $\Delta_d(f)$ obtained earlier in [11–15] at various temperatures and dislocation densities, the curves presented in Fig. 1 have a significant difference – their high-frequency asymptotes almost overlap. This effect characteristic of the radiation pinning of dislocations alone was also observed by Stern and Granato [6] with irradiated copper samples when verifying their theoretical estimates. Some spread of the high-frequency asymptotes (see Fig. 1) most probably appeared due to insignificant errors accompanying acoustic measurements or in the process of description of experimental data by a theoretical pattern [8, 16].

The effect of a shift of the frequency spectra $\Delta_d(f)$ with increase in the irradiation dose is demonstrated in Fig. 2 by experimental curves *1* and *2* for the maximum decrement Δ_m and the resonance frequency f_m, respectively. As one can see, the indicated resonance characteristics change monotonously and synchronously, though in the opposite directions.

According to [8], the following expression is valid for the decaying branch of the resonance curve $\Delta_d(f)$:

$$\Delta_\infty = \frac{4\,\Omega\, G\, b^2 \Lambda}{\pi^2 B\, f}, \tag{1}$$

where Δ_∞ denotes the dislocation decrement for the frequencies $f \gg f_m$, Ω is the orientation factor taking into

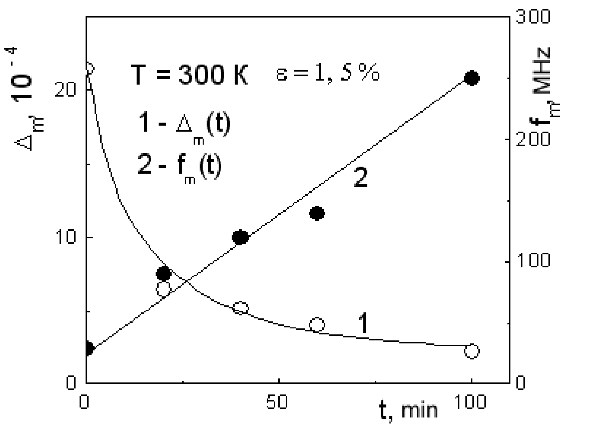

Fig. 2. Decrement Δ_m (*1*) and the resonance frequency f_m (*2*) as functions of the irradiation time of LiF crystals

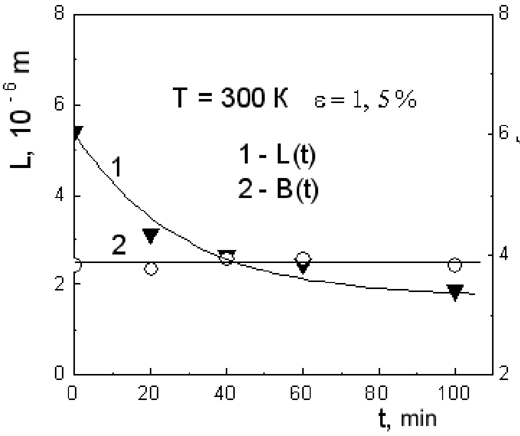

Fig. 3. Dislocation segment length (*1*) and the dislocation damping coefficient (*2*) as functions of the irradiation time of LiF crystals

account that the reduced shear strain in the slip plane is lower than the strain applied to the sample, G is the shear modulus of the acting slip system, b is the absolute value of the Burgers vector, Λ is the dislocation density, and B is the damping constant.

The calculation formulas for the position of the resonance maximum resulting from the theory proposed in [8] have the form

$$\Delta_m = 2,2\Omega\,\Delta_0\,\Lambda\,L^2, \quad f_m = \frac{0,084\,\pi\,C}{2\,B\,L^2}\,, \tag{2}$$

where L stands for the mean effective length of the dislocation segment, $\Delta_0 = (8Gb^2)/(\pi^3 C)$, C is the effective tension of a bent dislocation calculated as $C = 2Gb^2/\pi(1-\nu)$, and ν is the Poisson's ratio.

Using the high-frequency asymptotes of the dependences $\Delta_d(f)$ measured in this work and the data set for the parameters G, b, ν, Ω, and Λ, the absolute values of the parameters B and L in the range of the irradiation times 0–100 min were calculated using formulas (1)–(3). The values of the other physical characteristics used in these calculations were borrowed from recent work [12]: $G = 3.53\times10^{10}$ Pa, $b = 2.85\times10^{-10}$ m, $\nu = 0.273$, and $\Omega = 0.311$. The dislocation density $\Lambda = 3.43\times10^{10}$ m^{-2} for $\varepsilon = 1.5\%$ was determined from the graph $\Lambda(\varepsilon)$ [12].

Figure 3 presents the mean effective length of the dislocation segment L (curve *1*) and the dislocation damping coefficient B (curve *2*) for LiF crystals preliminarily deformed to $\varepsilon = 1.5\%$ as functions of the duration of X-ray irradiation of the samples at $T = 300$ K in the interval 0–100 min. The form of curve *1* clearly demonstrates that the quantity L reaches its maximum value in the case of unirradiated samples and monotonously decreases with increase in the radiation dose. For the highest dose equal to 660 R, the parameter L decreased approximately threefold as compared to its maximum value. The authors of works [1, 6] considered that this effect is a clear evidence of the fact that damages in crystals arising during their irradiation must play a role of pinning centers for mobile dislocations. When constructing curve *2* (Fig. 3), it turned out that the viscosity coefficient B for the LiF crystals under study does not depend on the irradiation of the samples with doses 0–660 R. This fact confirms the opinion stated in [17] that the magnitude of the dynamic dislocation drag represents a fundamental characteristic of a crystal and its level is determined only by the interaction of mobile dislocations with the phonon subsystem of the studied sample, which is also proved by a cycle of works [11–15]. Comparing the experimental results on LiF obtained in this work and reported in [10], one can notice that, in the both cases, the X-ray irradiation of the crystals results in a decrease of the amplitude of the frequency curves $\Delta_d(f)$ and their shift toward higher frequencies. As concerns the absolute estimates of B obtained for irradiated samples in the compared studies, there arises a significant disagreement. It is unclear which factors could result in the obtaining of the coefficient B increased by a factor of 1.5 at the constant parameters T and Λ, which is reported in [10]. According to the results presented in Fig. 3, the irradiation induces only changes in the parameter L that does not at all influence the damping constant B (see expression (1)). The assumptions made by the authors of [10] that the coefficient B can increase due to the redistribution of phonons located in the vicinity of radiation defects seems to be groundless, at least, under the conditions of small irradiation doses.

4. Conclusions

1. The effect of X-ray irradiation with doses in the interval 0–660 R on the localization of the damped dislocation resonance in LiF single crystals preliminarily deformed to $\varepsilon = 1.5\%$ was investigated by the pulsed technique at the temperature $T = 300$ K and in the frequency range 22.5–232.5 MHz. It is established that a gradual increase in the irradiation dose of the crystals induces two effects – a monotonous shift of the parameters of the resonance maximum toward higher frequencies and the damping of the dislocation resonance amplitude.

2. The independence of the dynamic dislocation drag coefficient B of the irradiation dose in the range 0–660 R for LiF crystals was first discovered. The established independence of the parameter B of the irradiation time in the interval 0–100 min confirms the known ideas that the dynamic dislocation drag at high temperatures ($T = 300$ K) is mainly determined by dissipative processes in the phonon subsystem of a crystal. As regards the contribution made to the dislocation drag by the term caused by the possible interaction mechanism "dislocation – radiation defect", it was not registered under the considered experimental conditions.

3. The dependence of the mean effective length of dislocations on the irradiation time was first determined for LiF crystals. The reduction of the parameter L with increase in the irradiation dose is explained by the intensified locking of mobile dislocations by pinning centers appearing in the process of irradiation of crystals. Based on the obtained dependence, the monotonous shift of the parameters of the resonance maximum in the indicated range of irradiation doses is explained.

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АКУСТИЧНІ ДОСЛІДЖЕННЯ ВПЛИВУ РЕНТГЕНІВСЬКОГО ОПРОМІНЕННЯ НА ДИНАМІЧНЕ ГАЛЬМУВАННЯ ДИСЛОКАЦІЙ У КРИСТАЛАХ LiF

Г.О. Петченко

Р е з ю м е

Імпульсним методом в інтервалі доз опромінення 0–660 рентген в області частот 22,5–232,5 Мгц при кімнатній температурі досліджено дислокаційний резонанс в монокристалах LiF із залишковою деформацією $\varepsilon = 1,5\%$. На основі аналізу отриманих даних було встановлено, що в умовах опромінення кристалів істотно змінюється лише частотна і амплітудна локалізація дислокаційного резонансу внаслідок змінення середньої ефективної довжини дислокаційного сегмента, але величина коефіцієнта в'язкості B залишається незмінною.