

METHODS FOR DETERMINING THE SPECIAL STATE OF THE DISLOCATION STRUCTURE IN ALKALI-HALIDE CRYSTALS

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Three methods of the critical value of plastic deformation ε^* determining, at which the mechanisms of plastic deformation of alkali-halide crystals (AHC) change, are considered. In the first method, selective etching of crystals is used, in the second – measurements of amplitude-independent internal friction with establishment of the dependence of the average effective length of the dislocation segment on the dislocations density, and in the third – establishing of the radiation attenuation spectral index under the influence of deformation and x-irradiation, as well as the dynamics of the absorption bands formation, which is due to the appearance of a volumetric concentration of color centers. It is shown that the optical absorption method is the most attractive from the point of view of obtaining of high-precision results at the same time it is the most convenient to use in experimental technology.

INTRODUCTION

Alkali-halide crystals are actively used in devices for various purposes [1–4]. For the nuclear power industry, this is an elementary base of radiation diagnostic devices, dosimetry, plasma diagnostic systems (UV windows), the basis of scintillators and lasers at color centers. The latter are used in nuclear power both for remote dosimetry (remote reading of the absorbed radiation dose by AHC-based detectors) and effective impact on UV windows (remote destruction of color centers in windows and removal of the “darkening” effect). In addition, these crystals are model, and it was on them that most of the data that formed the basis of the theory of radiation strength physics was obtained.

In the practical use of AHC, their dislocation structure is important, in particular, the critical value of plastic deformation ε^* , at which the mechanisms of plastic deformation change. As the deformation increases in the section $\varepsilon < \varepsilon^*$, mobile dislocations detach from point stoppers – impurities, radiation defects. At the transition point ε^* , the detachment of dislocations is maximal. When ε exceeds ε^* , the dislocations within the active slip systems interact with the forest dislocations, leading to their reattachment at strong obstacles such as the nodes of the dislocation grid [5]. At the point ε^* , the mobile dislocations of the primary slip planes exhaust the possibilities of their movement and the movement in the secondary slip planes begins. That is, for structural elements based on AHC, the critical point ε^* is important.

If AHC are used as a basis for delay lines of electric signals in device circuits, then with residual deformation ε^* the advantages of AHC as materials with weak absorption [5] are significantly reduced. In this case, the absorption of ultrasound in the material will be maximum, and the delay line itself will be the least

effective. That is, it is necessary to monitor that the deformations in this case do not reach the value of ε^* .

If AHC are used for optics (lenses, prisms, optical windows, light filters), then the presence of residual deformation of the value ε^* will cause minimal transmission of radiation in this material. This can be useful in conditions where the optical element plays the role of a correction filter [6] and its function consists precisely in a special correction for the amplitude of the radiation passing through it. That is, for optical elements, the dislocation state of the material is also critically important.

It is fundamentally important to achieve the critical value of plastic deformation ε^* in the case when AHC is used as the basis of lasers on color centers. The coloring centers enrich the energy spectrum of the crystal, and this is exactly what is used in lasers with a tuned wavelength (a broad spectrum of laser wavelengths and the ability to impact various materials). Here, on the contrary, at the residual deformation ε^* , the lasers are maximally effective. Precisely because ε^* is characterized by the maximum liberation of dislocations from point defects, the maximum number of free resonators – coloring centers, which expand the energy spectrum of the crystal used as a laser – appears in the volume of the crystal. At $\varepsilon < \varepsilon^*$ and at $\varepsilon > \varepsilon^*$, the concentration of color centers in the crystals is lower, because the dislocations act as traps for radiation defects, which reduces the efficiency of the AHC-based laser.

Thus, the determination of the critical value of ε^* is important for the use of ionic crystals as structural elements of devices, and the presence of a dislocation structure caused by deformation can bring both negative and positive effects.

The purpose of this work is to review the experimental methods of determining ε^* and to

demonstrate the non-standard use of the optical absorption method to solve this particular problem.

1. MICROSTRUCTURAL STUDIES USING SELECTIVE ETCHING OF CRYSTALS

The relationship between the dislocation density Λ and the deformation ε was determined in alkali-halide ionic crystals of KBr using a selective etching method, as illustrated in Fig. 1.

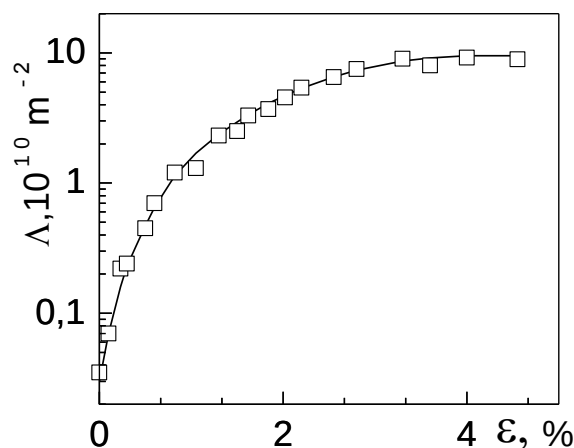


Fig. 1. Dependence of the dislocation density Λ on the magnitude of the preliminary deformation ε in KBr crystals

Fig. 1 shows a wide section ($\varepsilon \sim 1...2.4\%$) of the inflection of the $\Lambda(\varepsilon)$ curve, which characterizes the transition from primary to secondary slip systems during the movement of mobile dislocations. A fairly wide region of the inflection causes a too “blurred” critical value of ε^* .

Additionally, this method is linked to numerous experimental studies because of its reliance on selective crystal etching, which diminishes its overall appeal. For correct counting of etching figures, a special developed technological procedure should be used [7]. The essence of this procedure is to select a special mode of slow crystal deformation, as a result of which dislocations evenly fill the volume of the crystal. At the same time, sliding bands, which complicate the process of counting etching figures and introduce the main error into this method, do not occur. This methodology ensures the measurement of the curve $\Lambda(\varepsilon)$ with sufficient accuracy, but the critical value of the plastic strain ε^* is determined with a large error.

2. DETERMINATION OF THE EFFECTIVE DISLOCATION SEGMENT LENGTH AS A FUNCTION OF DISLOCATION DENSITY

The best result was achieved in the second method. Its essence boils down to the sounding of crystals in the frequency range of 7.5...232.5 MHz and the correct establishment of the dependences on the density of dislocations of the average effective length of the dislocation segment $L(\Lambda)$, which clearly shows the inflection near the special point Λ^* , which corresponds to the value of ε^* .

Fig. 2 shows the dependences of $L(\Lambda)$ obtained at different temperatures in the range of 77...300 K by the amplitude-independent internal friction method.

For any of the temperatures in the given range of 77...300 K, the behavior of the $L(\Lambda)$ curve is the same – a gradual increase in the length of the dislocation segment, due to the detachment of dislocations from “weak” point stoppers (present impurities), reaching the maximum detachment (achieving the critical value Λ^* at ε^*), and, subsequently, a gradual decrease in L due to anchoring by “strong” stoppers – dislocations “forest” [8].

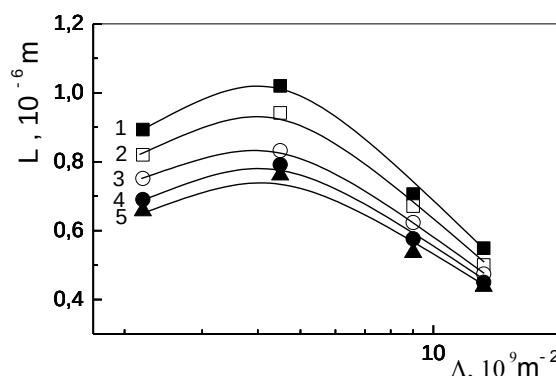


Fig. 2. Dependence of the average effective length of the dislocation segment L on the dislocation density Λ in KBr crystals at temperatures: 1 – 300 K; 2 – 250 K; 3 – 200 K; 4 – 150 K; 5 – 77 K

Increasing temperature variation in the considered interval only strengthens the observed effect. Moreover, the effect of detachment from “weak” stoppers is more strongly activated with increasing temperature, and the effect of attachment by “forest” dislocations is less pronounced, which indicates a weaker temperature dependence of the interdislocation interaction processes.

The main significance of the given curves is the clearly identified inflection of the $L(\Lambda)$ curves. It is from such curves that the values of Λ^* and ε^* can be determined quite accurately. This method is accurate and reliable, but its spread is limited by the need to use original non-standard equipment.

3. ASSESSMENT OF THE SPECTRAL INDEX OF RADIATION ATTENUATION INFLUENCED BY DEFORMATION AND X-RAY EXPOSURE

Work [9] studied the effect of deformation in the range of 0.155...3.3% and x-irradiation in the dose range of 0...1057 R on the dynamics of the formation of absorption bands due to the appearance of F-centers in high-purity LiF single crystals. For this purpose, the transmission spectra of $\tau_i(\lambda)$ in the mentioned crystals in the wavelength range of 220...650 nm were investigated.

In all studied LiF samples at an irradiation dose of 1057 R (Fig. 3), absorption bands were formed with the localization of the maximum on the axis of wavelengths at $\lambda_{max} \approx 248$ nm, which correspond to the formation of F-centers in the crystals. The dependences of $\tau_i(\lambda)$ change with deformation non-monotonically – they first decrease in amplitude in the interval 0.155...0.8% and, after passing the limit value of 0.8%, return to the initial state.

From the acoustic experiment conducted on these same samples, it is known that $\varepsilon^* = 0.8\%$ is precisely the boundary that separates the region of detachment of dislocations from stoppers during their movement along the primary slip planes from the region of reattachment of dislocations by “forest” dislocations [9].

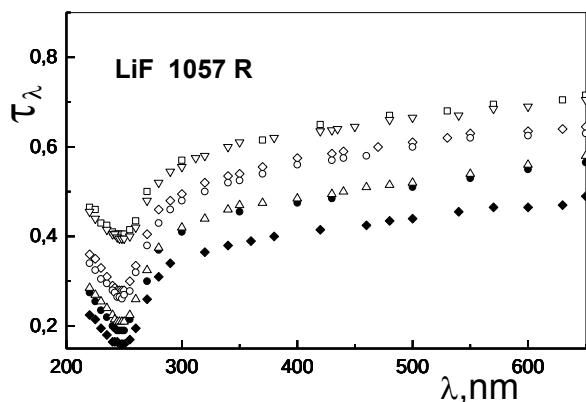


Fig. 3. Transmittance spectra of $\tau_\lambda(\lambda)$ in LiF crystals with a dose of x-irradiation of 1057 R for different values of the preliminary deformation of the samples: ∇ – 0.155%; $\diamond$ – 0.4%; $\bullet$ – 0.65%; $\blacklozenge$ – 0.8%; $\triangle$ – 1.2%; $\circ$ – 1.5%; $\square$ – 3.3%

Thus, $\varepsilon^* = 0.8\%$ is a reference point for both optical and acoustic (in the frequency range 7.5...232.5 MHz) experiments.

Given this fact, within the framework of Smakula's theory [10, 11], the dependence of the spectral attenuation index K_m on the magnitude of the previous deformation ε was determined (Fig. 4). The interaction of dislocations with centers of radiation origin was considered specifically for absorption bands, because the qualitative and quantitative composition of these defects is clear for them. Fig. 5 shows the dependence of the volume concentration of F-centers in LiF N_F crystals as a function of ε [9].

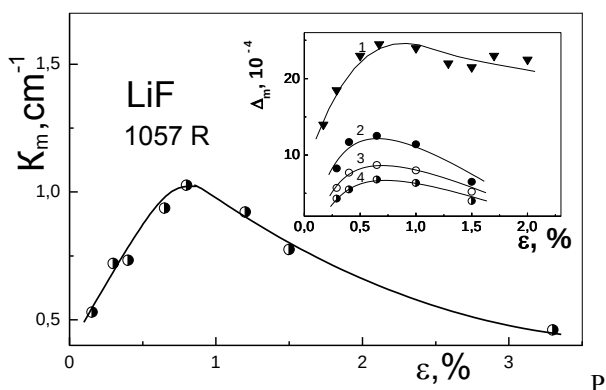


Fig. 4. Dependence of $K_m(\varepsilon)$ for LiF crystals subjected to an x-irradiation by dose of 1057 R.

Inset – acoustically obtained dependences on the deformation of the maximum dislocation decrement of ultrasound attenuation: 1 – non-irradiated LiF samples; 2–4 – crystals irradiated to different doses [12]

Fig. 4 illustrates an analogy in the behavior of both ultrasound dislocation absorption and the peak of electromagnetic wave attenuation caused by deformation – both occurring near the critical strain

value $\varepsilon^* = 0.8\%$. The nature of the behavior of the $K_m(\varepsilon)$ and $\Delta_m(\varepsilon)$ curves reverses at this point. Regarding the behavior of the $\Delta_m(\varepsilon)$ curves, the physics of the process is also explained by the detachment of dislocations from “weak” stoppers, the maximum release of dislocation segments at $\varepsilon^* = 0.8\%$ and their reattachment by “strong” stoppers with further increase of ε [12].

As for the behavior of the $K_m(\varepsilon)$ curve, here the detailing is given by the dependence on the deformation of the volume concentration of N_F coloring centers, presented in Fig. 5.

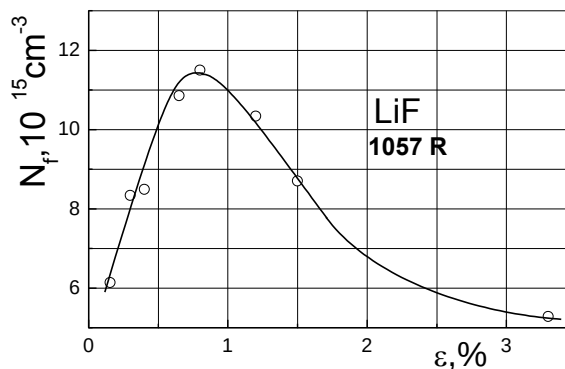


Fig. 5. The relationship between the concentration of F-centers and the strain $N_F(\varepsilon)$ in LiF crystals subjected to an x-irradiation dose of 1057 R

As the strain increases in the range of 0.155...0.8%, the dislocations “reset” the F-centers, their number in the volume of the crystal, as free resonators capable of resonantly responding to the electromagnetic excitation of the crystals, increases accordingly, as a result of which $K_m(\varepsilon)$ increases. At the point $\varepsilon^* = 0.8\%$, we observe the maximum number of F-centers, and, accordingly, the maximum attenuation of the electromagnetic wave (minimum transmission – see Fig. 3). Then, when $\varepsilon > \varepsilon^*$, it is the interdislocation interaction that is activated, the dislocations of the active slip planes are reattached by the “forest” dislocations, their mobility decreases, and, therefore, the F-centers begin to return to the impurity atmosphere of dislocations. The increase of F-centers on dislocations causes, accordingly, their decrease in the volume of crystals, which, in turn, is reflected in the decrease of the attenuation index (see Fig. 4) and the restoration of the permeability of crystals (see Fig. 3).

The accuracy of ε^* determining using the optical method is no worse than that of the acoustic method, but there is a significant advantage – it does not require non-standard measuring devices. Standard experimental equipment is sufficient for its implementation: a deformation machine, a source of x-irradiation and a spectrophotometer.

CONCLUSIONS

1. Based on the analysis of the deformation dependences of the density of dislocations $\Lambda(\varepsilon)$ and the average effective length of the dislocation segment $L(\Lambda)$ in KBr crystals, the spectral index of radiation attenuation $K_m(\varepsilon)$ and the volumetric concentration of color centers $N_F(\varepsilon)$ in LiF crystals, conclusions are

drawn regarding the possibility of determining the value of ε^* .

2. The value of ε^* is important for the structural elements of devices based on AHC, since when $\varepsilon = \varepsilon^*$ the dislocations of the active sliding systems are as free as possible from existing stoppers – impurities, radiation defects and dislocations of the forest.

3. As a result of the analysis, the following can be stated.

The method of ε^* determining from the dependence of the dislocation density Λ on the value of the previous deformation, obtained on the basis of microstructural studies using selective etching of crystals, is ineffective, and its main drawback is insufficient accuracy.

Determination of ε^* based on an acoustic experiment within the framework of the amplitude-independent internal friction method has sufficient accuracy. The disadvantage of this method is the difficulty of acoustic experiments conducting within the framework of amplitude-independent internal friction due to the need to use special original equipment.

The optical absorption method for determining ε^* is the most optimal for wide use, because on the one hand it allows you to determine ε^* with an accuracy no worse than that of the acoustic method, and on the other hand, it does not require the presence of original precision acoustic equipment and allows you to determine ε^* quite reliably on the equipment present in the vast majority of scientific laboratories.

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МЕТОДИ ВИЗНАЧЕННЯ ОСОБЛИВОГО СТАНУ ДИСЛОКАЦІЙНОЇ СТРУКТУРИ В ЛУЖНО-ГАЛОЇДНИХ КРИСТАЛАХ

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Розглянуто три методи визначення критичного значення пластичної деформації ε^* , при якому відбувається зміна механізмів пластичного деформування лужно-галогідних кристалів (ЛГК). У першому методі використовується вибіркове травлення кристалів, у другому – вимірювання амплітудно-незалежного внутрішнього тертя із встановленням залежності від густини дислокацій середньої ефективної довжини дислокаційного сегменту, а в третьому – встановлення спектрального показника ослаблення випромінювання при впливі деформації і х-опромінювання, а також динаміки утворення смуг поглинання, що обумовлено появою об'ємної концентрації центрів забарвлення. Показано, що найбільш привабливим із погляду одержання високоточних результатів одночасно з найзручнішою до використання технологією експерименту є саме оптичний абсорбційний метод.