Effect of crystal pre-straining on phonon damping of dislocations

A.M.Petchenko, G.A.Petchenko

Kharkiv National Academy of Municipal Service,
12 Revolutsii St., 61002 Kharkiv, Ukraine

Received June 18, 2008

The damped dislocation resonance in KBr crystals at 0.75 % residual strain has been studied in the 77 to 300 K temperature range using the pulsed echo technique. The sample strain change has been found to influence considerably the dislocation resonance localization but does not affect the absolute value of viscosity coefficient B and its temperature dependence. Basing on the frequency spectra and temperature dependences of other quantities, the temperature-dependent variations of the dislocation damping factor, B, and the average effective length of the dislocation segment, L, were determined within the mentioned temperature range. The dislocation fixation process in the crystals at 0.75 % residual strain at temperature lowering has been found to be controlled by the simultaneous action of weak and strong stoppers. Comparing the experimental $B(T)$ dependence with theoretical curve calculated using the Alshits-Indenbom theory, it has been established that the viscous dislocation damping in KBr crystals is limited by superposition of phonon wind and "slow" phonon relaxation mechanisms.

Импульсным эхо-методом исследован задемпфированный дислокационный резонанс в кристаллах KBr с остаточной деформацией 0,75 % в температурном интервале 77–300 К. Обнаружено, что изменение деформации образца существенно влияет на локализацию дислокационного резонанса, однако не сказывается на абсолютной величине коэффициента вязкости B и его температурном ходе. Из анализа частотных спектров и температурного хода других величин определены температурные изменения коэффициента демпфирования дислокаций B и средней эффективной длины дислокационного сегмента L в указанном интервале температур. Выявлено, что процесс закрепления дислокаций в кристаллах с остаточной деформацией 0,75 % при снижении температуры контролируется одновременным действием слабых и сильных стопоров. В результате сравнения экспериментальной зависимости $B(T)$ с теоретической кривой, вычисленной по теории Альшица-Инденбома, установлено, что вязкое торможение дислокаций в кристаллах KBr лимитируется суперпозицией механизмов фононного ветра и релаксации "медленных" фононов.

During last two decades, the phenomena being demonstrated as displacements of individual dislocations in ionic crystals (e.g., the magneto-plastic effect) are under a rather intense investigation using the modern experimental techniques [1]. The authors have revealed a high sensitivity of that effect to mechanical loading, electric field, and X-rays. The damped dislocation resonance being studied in this work is extremely sensitive to the above-mentioned factors. This work continues the studies of the KBr crystal pre-straining effect on the value and temperature dependences of phonon dislocation damping that were onset in [2–5]. In particular, the anomalous shift of the dislocation resonance maximum both in amplitude and frequency has been revealed. The information on acousto-mechanical, damping, and elastic characteristics of the crystals is of great importance both in designing and exploitation of functional electronic devices and in development of functional materials with pre-specified performance characteristics.

The purpose of this work is to study in experiment the phonon dislocation damping and the specific features of the dislocation resonance in KBr crystals pre-strained by compression up to 0.75 % in the 77–300 K temperature range. Such experiments are carried out as a rule using high purity crystals with fresh dislocations introduced thereto by straining. Each sample is pre-strained to not only cause the dislocation resonance but also to provide its frequency shift into a range convenient for observation. In [3], during the acoustic-mechanical experiments on KBr crystals, an anomalous shifting of the dislocation resonance maximum both in amplitude and frequency was revealed. That maximum was established to shift first towards lower frequencies and then, after stoppage, to move in opposite direction at a constant increase of the sample strain. Basing on frequency dependences $\Delta_d(f)$ (where Δ_d is the dislocation decrement of ultrasound damping; f, the ultrasound frequency), the dislocation damping factor B was found to be independent of the dislocation density Λ at $T = 300$ K. Along with the established functional dependence $B(\Lambda)$, the author [3] has obtained the absolute value of viscosity coefficient B in conditions eliminating the error admissible at the counting the etch pits. To obtain such results at temperatures lower than room one, KBr crystals with residual strains of 0.5 and 1 % were used in [4, 5]. The strains were selected intentionally to provide (according to guidelines from [3]) the limiting shifts of the resonance maximum in the $\Delta_d(f)$ curve towards lower and higher frequencies, respectively. According to [3], such a strain causes a stoppage in the maximum shift towards low frequencies that is changed to its movement in the opposite direction. It is expected that basing on the data obtained in this work, the problem of possible effect of the dislocation density Λ on the damping constant B value and its temperature dependence.

To study the temperature dependence of the damping coefficient, $B(T)$, a home-made measuring setup was used that provided the simultaneous measurements of the crystal acousto-mechanical characteristics at various straining rates within wide temperature and frequency limits using the waves varying in polarization and amplitudes [2–5].

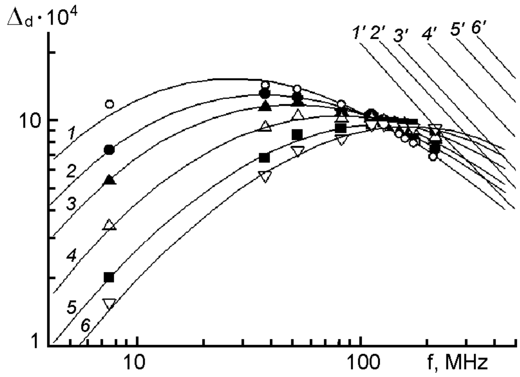

Fig. 1. Frequency dependences of dislocation decrement for KBr samples with residual strain of 0.75 % at various temperatures T, K: *1* – 300, *2* – 250, *3* – 200, *4* – 150, *5* – 100, *6* – 77,. Solid lines correspond to theoretical curves [8] and their high-frequency asymptotes.

The frequency dependence of the ultrasound damping decrement $\Delta_d(f)$ was studied in the frequency range of 7.5 to 217.5 MHz and temperature range of 77 to 300 K for KBr single crystals using the calibrated exponential curve superposition. To compare correctly the results from this work and those from the preceding ones, a series of 30 mm long samples having 18×18 mm^2 cross-section was used cleaved out from the same large single crystal and processed using the same technology as in [3–5]. The non-parallelism of the sample working faces was ±1 μm/cm. The fresh dislocations were introduced by compressing the sample along its long axis in the <100> direction coincident with the sounding one using an INSTRON testing machine at the deformation rate of about 10^{-5} s^{-1}. The samples were pre-strained up to 0.75 % prior to measurements.

Fig. 1 presents the experimental data on the damped dislocation resonance for KBr samples at the 0.75 % residual strain. The Figure shows that the frequency position and height of the resonance maximum vary with temperature. As the temperature is lowered, the dislocation loss decreases and the resonance curves are shifted monotonously towards higher frequencies. Consideration of Fig. 1 evidences that the measured dislocation absorption as a function of frequency, $\Delta_d(f)$, has the damped resonance character [7]. The experimental values are described well by a normalized frequency profile calculated for the exponential distribution of the dislocation loop lengths [8]. The theoretical and experimental curves were compared using numerical methods, the experimental curve being pre-transformed into log coordinates, $\lg\Delta_d(\lg f)$. According to recommendations [9, 10], the theoretical curve was fitted to experimental data only under orientation to the experimental points belonging to the descending experimental curve branch and in the resonance region. According to [7], the relationships describing the maximum position and the descending resonance curve branch are as follows:

$$\Delta_m = 2.2\Omega\Delta_0\Lambda L^2, \quad (1)$$

$$f_m = \frac{0.084\pi C}{2BL^2}, \quad (2)$$

$$\Delta_\infty = \frac{4\Omega G b^2 \Lambda}{\pi^2 B f}. \quad (3)$$

Here, Δ_m and f_m are the decrement and frequency values in the maximum; Δ_∞, the decrement value for $f >> f_m$; Ω, the orientation factor; Λ, the dislocation density; L, the average effective length of dislocation segment; $\Delta_0 = (8Gb^2)/(\pi^3 C)$, C being the effective stretch of bent dislocation ($C = 2Gb^2/\pi(1 - \nu)$); ν, the Poisson coefficient; G, the shear modulus of the acting slip system; b, the Burgers vector module. Using the obtained resonance curves, the B value can be calculated from the Eq. (3), the Δ_∞ being previously determined from the high-frequency asymptote and Λ by counting the etch pits. The correct estimation of the viscosity coefficient B requires obviously the accurate values of other parameters included in (3).

In this work, the temperature dependences of Ω and G parameters were used determined before for unstrained KBr single crystals [2, 11] at the acting slip system {110} <110>. To calculate the temperature dependences $\Omega(T)$ and $G(T)$, the formulas $\Omega = (C_{11} - C_{12})^2 / 4C_{11}G_{110})$ [12] and $G_{110} = 0.5(C_{11} - C_{12})$ [13] were used in [2, 11] with substituted elastic moduli C_{ik} taken from [2, 11]. The temperature-depended variation $b(T)$ of the b parameter were determined using the equation $b = a/\sqrt{(2)}$ [13] taking into account the thermal-induced changes in the lattice constant, $a(T)$ [14]. A special attention was given to reduction of errors in Λ parameter and monitoring of its possible variations during the experiments. For the selected sample geometry and its deformation direction, the slipping in the crystal

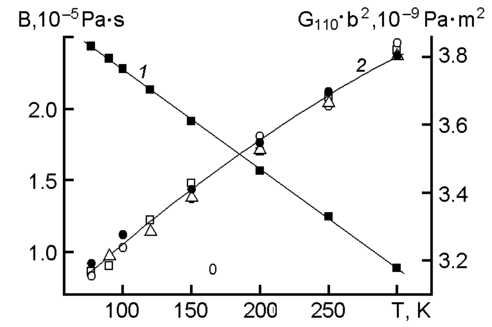

Fig. 2. Temperature dependences of Gb^2 parameter (*1*) and the dislocation damping constant (*2*). In Curve *2*, experimental points are taken from [2], (□) [4] (•), [5] (Δ), this work data (°) for KBr crystals with $\varepsilon = 0.75$ %.

occurs in four equally stressed slip systems {110} <110>. The dislocations were revealed by selective etching of freshly cleaved {100} surfaces parallel to the crystal long axis. To determine the dislocation density, 100 to 150 fields were selected characteristic of various areas of the crystal faces. For the samples with residual strain of 0.75 %, the average Λ value, as determined by the etch pit counting, has been found to be 9×10^9 m^{-2}. The absolute Λ value has been determined at an accuracy of 15 to 20 %. This was attained by using the slow crystal loading procedure [15] resulting in homogeneously distributed etch patterns arising on the crystal surface without slipping strips.

The consideration of the dislocation structure has shown that the dislocation density in the strained crystal remains the same both prior and after the measurements mentioned. This is confirmed also by the hysteresis absence in the $\alpha(T)$ curves (α being the ultrasound absorption) measured at the sample cooling and heating. Indeed, a longitudinal wave directed along the sample long axis has the shearing stress components in the same slip planes as the applied compression stress. Thus, it interacts with dislocations being in those planes. In such conditions, the ultrasound damping should reflect the changes in the dislocation structure parameters (the dislocation density Λ and the average dislocation segment length L). A change in Λ should obviously result in a hysteresis loop in the $\alpha(T)$ curve, that is not the case in experiment.

Fig. 2 shows the temperature-dependent variations of the Gb^2 parameter and the coefficient B of the dislocation viscous damping. It is seen that Gb^2 increases and B drops as the testing temperature decreases.

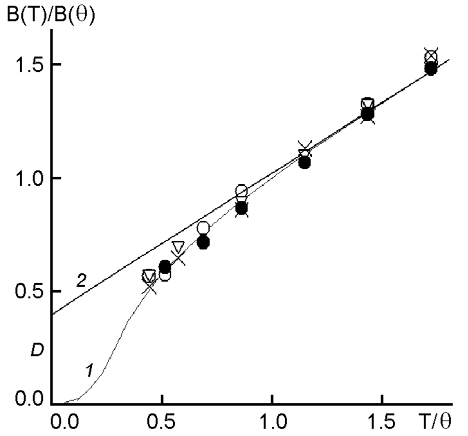

Fig. 3. Comparison of $B(T)$ dependences according to Eq. (4) (curve *1*) with experimental data for a KBr crystal: °, data taken from [2]; ∇, ×, •, data from this work for KBr crystals with ε 0.5; 0.75; and 1 %, respectively. Curve *2*, high-temperature asymptote; *D*, theoretical parameter [16] used in Eq. (4).

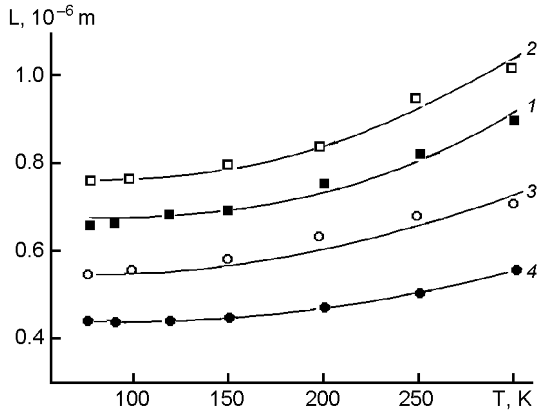

Fig. 4. Temperature dependences of effective dislocation segment length L for KBr crystals at different dislocation densities Λ, 10^9 m^{-2} : *1* – 2.2, *2* – 4.5, *3* – 9.0, *4* – 13.0.

The experimental B values for samples with various dislocation density Λ have been found to fall satisfactorily on one and the same curve, thus evidencing that coefficient B is independent of Λ parameter.

To explain the experimental $B(T)$ curve (curve *2* in Fig. 2), it was compared to theoretical one calculated using the expression [16]

$$\frac{B(T)}{B(\theta)} = \frac{f_1(T/\theta)}{f_1(1)}(1 - Df_2(1)) + D\frac{\theta}{T}f_2(T/\theta), \quad (4)$$

where D is a dimensionless parameter determined from experimental data by extrapolating to zero the high-temperature asymptote $B(T)/B(\theta)$ as a function of T/θ. The comparison was carried out using the Debye temperature $\theta = 174.5$ K calculated in [2]. The values of other functions included in [4] were taken from [16]. The comparison results at experimental value $D = 0.4$ are shown in Fig. 3. The theoretical curve is seen to describe well the experimental data. The agreement between theoretical and experimental $B(T)$ curves evidences that the viscous damping of dislocations in KBr crystals is limited by superposition of the "slow" phonon relaxation and phonon wind mechanisms.

Once the temperature dependence of B was established, it became possible to trace the temperature-dependent changes in the average dislocation segment length L. Its values were found using the relation (2) as $L = [0.084 \cdot G \cdot b^2 / \ B \cdot f_m \cdot (1 - \nu)]^{0.5}$ where the Poisson coefficient $\nu = C_{12}/(C_{11} + C_{12})$ [13] was determined from the values of elastic moduli C_{ik} calculated in [2, 11]. The temperature dependences $L(T)$ of the effective length of dislocation segment for crystals with different residual strains are presented in Fig. 4. The trend of L shortening at lowering temperature is seen to be conserved at any residual strain. However, at a continuous strain increase, the $L(T)$ curves are shifted in height non-monotonously. First, as the strain rises from 0.23 to 0.5 %, the $L(T)$ dependence is shifted towards larger L values (Fig. 4, curves *1* and *2*) and then, at 0.75 and 1 % strains, the shift sign is reversed (Fig. 4, curves *3* and *4*). Consideration of $L(T)$ curves shows that the inversion of the frequency spectra $\Delta_d(f)$ takes place not at $T = 300$ K but also at other temperatures within the 77 to 300 K range. A detailed consideration of those curves has revealed another feature of importance associated with variations in their slope. The L quantity has been found to decrease by approximately the same value (by a factor of 1.35) for the samples with residual strain of 0.23 and 0.5 % at the temperature decrease from 300 to 77 K. However, its relative shortening at the same temperature decrease for the samples with residual plastic strain of 0.75 and 1 % has been found to be smaller (by a factor of 1.31 and 1.25, respectively). The regularities revealed seem to be connected with two different manners of the dislocation segment pinning. At small strains (up to 0.5 %) when only primary slip planes are activated in the crystal, the dislocations are pinned at the temperature dropping by weak pinning centers, namely, by point defects of complexes thereof. As the strain increases (above 0.5 %), the dislocations of the primary slip plane start to interact with the forest dislocations arising in the secondary planes causing the formation of dislocation blocks being strong pinning centers [7].

In [2], the possible causes of dislocation pinning by stoppers and the experimentally observed shortening of dislocation segment length at the temperature decrease from 300 to 77 K were discussed in details. According to [2], after the sample pre-straining, a dislocation, being cleared out of stoppers, makes first forced vibrations in the ultrasound wave field. The vibratory strain amplitude of the wave was found to be 10^{-7}. At such temperatures, the dislocation cannot be pinned again due to high level of thermal fluctuations and low binding energy between the dislocation and impurities. The situation changes abruptly as the temperature decreases. Now the density of the elementary excitation gas starts to decrease while the dislocation binding energy with impurities and the linear tension force ($\sim Gb^2$) (Fig. 2) increase due to rising shear modulus G. Thus, the impurity atoms become real pinning points. These processes caused by the temperature lowering result in decreasing B values (Fig. 2) due to a substantial increase in Δ_∞ as compared to ΩGb^2 and the shortening effective length of dislocation segment (Fig. 4) caused by its pinning by "weak" stoppers unable to reveal themselves at higher temperatures. The revealed effect of decreasing changes in L at temperature lowering (Fig. 4, curves *3* and *4*) is due likely to strong stoppers arising in the crystals with 0.75 % and 1 % residual strains: in the presence of strong stoppers, the part played by weak ones in the dislocation segment pinning becomes less and less appreciable. At initial strains of 0.23 and 0.5 % when the forest dislocations are not appeared in the crystal, the effective segment length L and dislocation density Λ are still increased, that is manifested as the increasing dislocation resonance amplitude and its shift towards lower frequencies. If such crystals are then cooled, the shortening of L therein will be the same in spite of different dislocation structures. This is an evidence that as the temperature decreases, the dislocations in those crystals are pinned by weak pinning centers only.

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Вплив попереднього деформування кристалів на фононне гальмування дислокацій

О.М.Петченко, Г.О.Петченко

Імпульсним луна-методом досліджено задемпфований дислокаційний резонанс у кристалах KBr із залишковою деформацією 0,75 % у температурному інтервалі 77–300 К. Виявлено, що зміна деформації зразка істотно впливає на локалізацію дислокаційного резонансу, однак не позначається на абсолютній величині коефіцієнта в'язкості B і його температурному ході. З аналізу частотних спектрів і температурного ходу інших величин визначено температурні зміни коефіцієнта демпфування дислокацій B і середньої ефективної довжини дислокаційного сегмента L у вказаному інтервалі температур. Виявлено, що процес закріплення дислокацій у кристалах із залишковою деформацією 0,75 % при зниженні температури контролюється одночасною дією слабких і сильних стопорів. У результаті порівняння експериментальної залежності $B(T)$ і теоретичної кривої, обчисленої за теорією Альшиця-Інденбома, встановлено, що в'язке гальмування дислокацій у кристалах KBr лімітується суперпозицією механізмів фононного вітру і релаксації "повільних" фононів.