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THE COMPETITION OF MOTT AND FRIDEEL TYPE STOPPERS AS THE MAIN BLOCKING MECHANISMS IN MOBILE DISLOCATIONS OF KBr CRYSTALS

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The experimental data of pulsed-method research in the frequency range 7.5...232.5 MHz of the preliminary deformation effect ε in the range of 0.23…1%, and at the temperature interval 77...300 K on the frequency spectra localization $\Delta_d(f)$ in the dislocation decrement of ultrasonic attenuation, as well as on dynamic (B) and structural characteristics (L, Λ) of KBr crystals. The competition of strong and weak stoppers in the processes of blocking mobile dislocations in the investigated crystals is vividly traced. The limiting value of ε = 2.25% is established, above which the thermal activation of the mobile dislocations' detachment from Friedel type stoppers is completely masked by more efficient processes of fixing dislocations by dislocation network nodes (Mott stoppers), which gave us grounds to recommend performing of further studies in this direction.

INTRODUCTION

The temperature behavior of the coefficient B was attempted to study by many authors, both on metals, and on ionic crystals [1–11]. The overwhelming majority of these works were really performed in the form of observing the individual dislocations' mobility, slip bands, and also as the results of high-speed crystals' deformation. The most reliable method [1–3], that allows us to study the phonon-dislocation interaction in crystals, the consideration of which is directly based on the results of the temperature behavior study of the parameter B, is the method of amplitude-independent internal friction. In the works [12–17], the authors managed to achieve quite reliable results due to which the verification of the Granato-Lucke string dislocation theory [1] and the quantum-mechanical theory of dislocations' dynamic inhibition by Alshits-Indenbom [2] was performed. The results [12–17] showed that the dynamic inhibition of dislocations in the investigated KBr crystals in the range 77...300 K is limited by the superposition of two phonon mechanisms: the phonon wind and the relaxation of “slow” phonons. The influence of the samples' dislocation structure in the range of the dislocation density variable $\Lambda = 2{,}2\cdot10^9\ldots13\cdot10^9$ m^{-2} on the absolute value of B and its temperature course in the indicated thermal interval was also studied. It turned out that for temperatures at the interval from the nitrogen to the room one, constant of the dislocations' dynamic breaking B does not depend on the density of mobile dislocations Λ in the investigated crystals. The mentioned result is of utmost importance, since none of the laboratories in the world have studied the problem of the mobile dislocations' density influence on the temperature variation of the B value, and, hence, on the efficiency of the various phonon mechanisms' action in crystals. The solution to this problem and the experimental proof of the fact, that the dynamic braking temperature course of dislocations in crystals is unchanged in the variation of the parameter Λ, and it provides significant opportunities for the further development of the dislocation theory based on the generalization of experimental data obtained in different laboratories on identical crystals. The fact is that when preparing samples for research, all scientific schools use their own materials and experimental techniques, that's why there are some inconvenience when trying to consider the experimental results of various laboratories from a single angle of view – the samples' chemical composition differs greatly (the points of fixation at the dislocations), various methods, conditions and equipment for growing crystals (different levels of “growth” dislocations, different technological schemes of samples' annealing, speed modes' variability of deformation machines, etc.). That is, all samples have a different dislocation structure, own unique preparation “history”, and it is difficult to compare and evaluate them according to a single graduation. But works [12–17] showed that all these dissimilitudes are completely insignificant for the general picture of the dislocations' phonon braking in crystals.

In the view of the theory effectiveness verifying [1], the works [12–17] are useful, in general, because they were performed on the basis of an experiment, the formulation of which was carried out within the framework of the model [1]. String model [1] provides two types of pinning points – nodes of a dislocation grid (Mott stoppers) and weaker centers of fastening (Friedel stops). The removal from the Friedel stoppers (impurities, in our case) occurs due to thermal or mechanical (pre-deformation of crystals) activation. It was the competition of these mechanisms that determined the mobility of dislocations in our experiments.

The purpose of this paper is to generalize the results obtained in [12–17] for the boundaries of the model's efficiency settlement [1] and to describe physical processes of the dislocation mobility thermal activation in crystals.

MATERIALS AND EXPERIMENTAL TECHNIQUES

In works [12–17] we investigated the diminished dislocation resonance in single crystals of KBr with degrees of previous deformation ε = 0.23; 0.5; 0.75, and 1% in the temperature range 77...300 K using longitudinal waves in the frequency range 7.5...232.5 MHz. The precision measurements of the ultrasonic attenuation were performed by the method of exponential overlay on single crystals, oriented along the crystallographic direction <100> at the apparatus [18]. The presence of impurities in samples, detected by methods of chemical and spectral analysis, was (weight %): Cu ~ 10^{-6}; Mn, Ag ~ 10^{-5}; Pb, Mg, Al, Ti ~ 10^{-4}; Fe ~ 10^{-3}. From the crystals grown by means of the Kiropoulos method, prototype samples were made, the size of which was approximately 18x18x30 mm. The samples' parameters took into account the basic authors' recommendations [19–21] regarding to the choice of their geometry – the cross-section and the length of the crystal, which is subject to acoustic measurement. The flat-parity of the samples corresponded to the recommendations [20] and constituted ± 1 μm/cm, that was controlled by the optimeter IKB. The scheme of samples' annealing after machining to eliminate internal stresses was as follows: heating crystals in a muffle furnace MP-2UM to a temperature of ~ $0.8 \cdot T_m$ (for the investigated crystals – up to 600 °C), time elapsing at the given temperature, and slow cooling at a rate of ~ 10 °C/h. The duration of annealing was about 15 hours. The temperature was controlled by a chromel-aluminium thermocouple connected to a digital voltmeter V7-27. Technologies of deformation and low-temperature measurements of samples are minutely described in [12–17].

RESULTS AND DISCUSSION

The results of studies [12–17] on the influence of temperature in the range 77...300 K on the behavior of the frequency spectra of the dislocation absorption $\Delta_d(f)$ of ultrasound in specimens with different residual deformation values and the resulting data with respect to the comparison of the temperature velocity *B(T)* calculated within the framework of the Granato-Lucke theory [1] with the theoretical curve of Alshits-Indenbom [2], that is shown in Fig. 1.

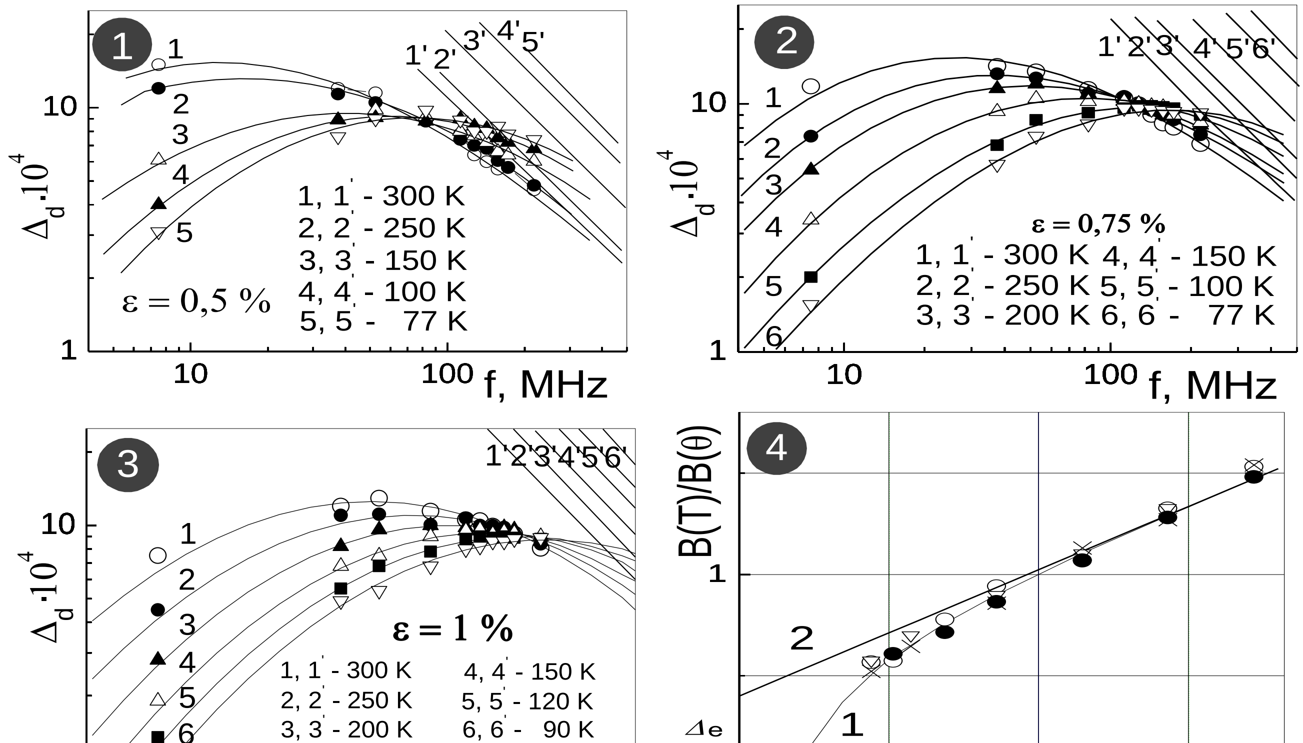

Fig. 1. Frequency dependences of the dislocation decrement for different temperatures of the interval 77...300 K (1–3). Solid curves – theoretical profiles (1–5) [1, 22]; straight lines (1'–5 ') – their high-frequency asymptotes; 4 – comparison of the combined temperature transition [2] B(T)/ B(θ) with experimental data for KBr crystal (o – data of papers [12, 13]; ▽, ×, • – data of works [14–17] for crystals of potassium bromide with deformations ε = 0.5; 0.75, and 1%); curve 2 – high-asymptote; Δ_e – phenomenological parameter of the theory [2]

One can see (see Figs. 1.1–1.3) that the experimental points for the curves $\Delta_d(f)$ are sufficiently well described by the theoretical frequency profile obtained by the authors [22] for the exponential distribution of the dislocation segments in length, which is mentioned in the theory [1]. Also, there is a tendency for the frequency spectrum to be offset both in frequency – to the direction of higher *f*, and in the amplitude – to the direction of smaller Δ_d when the temperature decreases at the studied interval. It is also obviously seen (see Fig. 1.4) that the variation in the samples' deformation within the range of 0.23...1% does not lead to changes in $B(T)$ and therefore does not affect the process of mobile dislocations' phonon braking in the investigated crystals. After the temperature After the temperature course establishing $B(T)$ it became possible to trace the thermal variations of the average effective dislocation segment length L. The value L could be determined with the help of the relations [1], minutely described in papers [12–17]. The family of curves $L(T)$ for KBr crystals with dislocation density within the interval of $\Lambda = (2.2…13)\cdot10^9\,m^{-2}$, corresponding to the deformations of the interval 0.23...1%, is shown in Fig. 2.

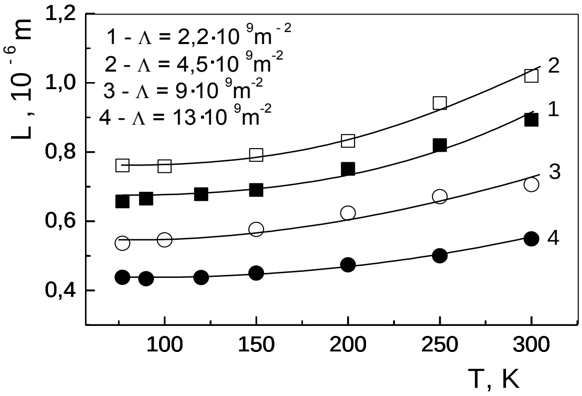

Fig. 2. The temperature dependences of the average effective dislocation segment length L for KBr crystals with different dislocation density

One can see that the tendency to reduce the average effective length of the dislocation segment with a decrease in temperature for any value of residual deformation of the sample is sustained. However, the of the dependences' displacement at the height $L(T)$ under conditions of deformation growth is non-monotonic. Initially, with an increase in deformation from 0.23 to 0.5%, we observe the displacement of $L(T)$ in the area of higher L values (see curves 1 and 2, Fig. 2, and then, at deformations of 0.75 and 1%, the offset sign changes to the opposite (see curves 3 and 4, Fig. 2). The given family of curves $L(T)$ proves that the inversion shift of the frequency spectra $\Delta_d\ (f)$ [12–17], caused by deformation of the crystal is observed for all temperatures of the interval 77...300 K. In a detailed examination of these curves, it was noted that the pace of their growth, along with the temperature, is different for the crystals with various dislocation density. For the crystals with deformations of 0.23 and 0.5% (see curves 1 and 2 in Fig. 2), the value L increases in the temperature range by 1.35 times. Curve 3 corresponding to KBr crystals with ε=0.75%, already has a slower slope with a temperature rise of 1.31 times, and curve 4 for KBr with ε = 1% shows the slightest increase in L – only 1.25 times. The presented tendencies have an expected physical explanation. In the course of minor deformations (up to 0.5%), only the primary sliding surfaces are activated in the crystal, and the dislocations have a noticeable effect of pinning with the weak stoppers – point defects. At 0.75%, in the crystal, secondary systems of sliding start to operate and the motion of the dislocations begins to be limited, except for the weak, stronger stops – the nodes of the dislocation grid start their functioning. Here, the effects of the dislocation interaction with the weak stoppers begin to become less noticeable against the background of a more effective dislocations' interaction effect. The further increase in deformation makes this grid of dislocations even more dense, so the effect of the dislocations' interaction with the point centers of blocking is masked to a greater extent. We can also assume that in a very strongly deformed crystal, the dependence of $L(T)$ can go almost parallel to the abscissa axis. This will correspond to a strongly dislocation crystal, whose length of the Franck grid is close to the size of the dislocation segment, limited by the point pinning centers.

Having processed our data within the framework of Origin 6.0, we came to the conclusion that the growth of ΔL of L value in the course of increase in the degree of crystals' KBr deformation, decreases according to the linear law $\Delta L = 1.45 - 0.2\cdot\varepsilon$. Thus, with the value of the crystals' preliminary deformation ε = 2.25% ΔL will be 1, that correspond to the zero growth of the dislocation segment along with the temperature increase. Therefore, we must state that the limit value ε = 2.25% defines the upper border of the deformation interval, in which it is possible, in principle, to study experimentally the competition of the Mott and Friedel stoppers' action in the course of the dislocations blocking [1, 23, 24]. Above this limit the dislocations are fixed by the nodes of the Franck grid, so that the process of their detachment is not thermally activated in the range of the studied temperatures 77...300 K.

It is most likely to fail checking this experimentally. If we recall that at $\Lambda = 13\cdot10^9\ m^{-2}$ (1%) the curves $\Delta_d(f)$, aimed for the low temperatures, have already begun to get out of the 7.5…217.5 MHz range limits with their falling branches. It can be predicted, that even if it will be possible to remove the frequency spectrum for a strongly deformed ($\varepsilon = 2.25\%$) crystal at room temperature, then it is not possible to speak of lower temperature studies.

The analysis can be useful for researchers studying the influence of various factors (irradiation [25–29], magnetic processing and variable chemical composition of samples [30–40]) on the mobility of dislocations in crystals. The preliminary deformation of the investigated samples is a mandatory procedure, since it allows for the insertion of mobile ("fresh") dislocations into the crystal, but the value of ε must not exceed the limit specified by us. Otherwise, all the mentioned investigated facts will be completely masked by the strong processes of blocking the mobile dislocations by the dislocations of the "forest".

CONCLUSIONS

On the basis of our analysis and generalization of the data obtained in previous studies on KBr crystals [12–17], it was established that the thermal activation of mobile dislocations' removing from Friedel-type stoppers of any physical nature is restricted by the limit values of the of the investigated crystals' previous deformation. According to our estimates, this value is ε = 2.25%. For ε, less than the specified limit value, the effect of thermal activation can be noticeable and, therefore, external factors influencing the dislocation structure of crystals (irradiation, magnetic processing, changeable chemical composition of samples) can be investigated by studying of the thermal effect on the unlocking of mobile dislocations by means of appropriate stoppers. For ε from 2.25% or more, fine structural experiments will become ineffective due to the strong background of dislocations' blocking by the dislocations of the “forest” and the masking of weak Friedel-type pinning points' effect by strong Mott-type stoppers [1].

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КОНКУРЕНЦИЯ МЕХАНИЗМОВ БЛОКИРОВКИ ПОДВИЖНЫХ ДИСЛОКАЦИЙ В КРИСТАЛЛАХ KBr СТОПОРАМИ ТИПА МОТТА И ФРИДЕЛЯ

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

Проанализированы экспериментальные данные исследований импульсным методом в области частот 7,5…232,5 МГц, а также их влияния предварительной деформации ε в диапазоне 0,23…1% и температуры в интервале 77…300 К на локализацию частотных спектров $\Delta_d(f)$ дислокационного декремента затухания ультразвука и на динамические (B) и структурные характеристики (L, Λ) кристаллов KBr. Отслежена конкуренция сильных и слабых стопоров в процессах блокировки подвижных дислокаций в исследуемых кристаллах. Установлено граничное значение ε = 2,25%, выше которого термическая активация процессов открепления подвижных дислокаций от стопоров фриделевского типа полностью маскируется более эффективными процессами закрепления дислокаций узлами дислокационной сетки (стопорами Мотта), что дало основание сделать рекомендации для дальнейших исследований в этом направлении.

КОНКУРЕНЦІЯ МЕХАНІЗМІВ БЛОКУВАННЯ РУХЛИВИХ ДИСЛОКАЦІЙ В КРИСТАЛАХ KBr СТОПОРАМИ ТИПУ МОТТА І ФРИДЕЛЯ

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Проаналізовано експериментальні дані досліджень імпульсним методом у межах діапазону частот 7,5…232,5 МГц, а також їх вплив попередньої деформації ε у діапазоні 0,23…1% і температури в інтервалі 77…300 К на локалізацію частотних спектрів $\Delta_d(f)$ дислокаційного декременту поглинання ультразвуку та на динамічні (B) і структурні характеристики (L, Λ) кристалів KBr. Простежено конкуренцію сильних і слабких стопорів у процесах блокування рухливих дислокацій в досліджуваних кристалах. Встановлено граничне значення ε = 2,25%, вище якого термічна активація процесів відкріплення рухливих дислокацій від стопорів фриделівського типу повністю маскується більш ефективними процесами закріплення дислокацій вузлами дислокаційної сітки (стопорами Мотта), що дало підставу визначити рекомендації для подальших досліджень у цьому напрямку.