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APPLICATION OF ACOUSTIC PULSE ECHO-METHOD FOR THE INVESTIGATION OF DYNAMIC AND STRUCTURAL DYSLOCATION CHARACTERISTICS OF CRYSTALS

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The multifunctional pulse equipment allowing to use the method of amplitude-independent internal friction in the frequency range 7.5 to 232.5 MHz is described. Equipment gives the possibility to investigate the peculiarities of the process of phonon-dislocation interaction in crystals, to carry out thermoactivation analysis of the process of dislocation of the dislocations about the stoppers under the action of temperature and elastic loading, to study processes of dislocation and mechanical relaxation in loaded samples in the quasi-elastic and plastic strain range.

INTRODUCTION

A lot of experimental and theoretical works [1-36]are devoted to the study of the dislocation dynamics and the processes of the interaction of dislocations with the stoppers of different physical nature, as well as with the gas of elementary excitations of the crystal by the methhod of amplitude-independent internal friction. To calculate the dislocation parameters, the damping coefficient *B* and the average effective length of the dislocation segment *L* in the studied crystals, there is a need to deteermine a number of parameters. The basic formulas for the calculations *B* and *L* according to the research of frequency spectra of the dislocation decrement of the extinction of ultrasound $\Delta_d(f)$ in the resonant and postreesonance ranges are as follows [1]: Resonance range:

$$B = \frac{7.48 \cdot 10^{-2} \cdot \Omega \cdot G \cdot b^2 \cdot \Lambda}{\Lambda \cdot f},$$
 (1)

$$L = \sqrt{\frac{0.084 \cdot G \cdot b^2}{B \cdot f_m \cdot (1 - \nu)}},$$
(2)

where $b = \frac{a}{\sqrt{2}}$ – the Burgers vector and $G_{<110>} =$

 $0.5 \cdot (C_{11} - C_{12})$ – the shear modulus (measure of resistance to the tangential load applied to the plane

<110>),
$$v = \frac{C_{12}}{C_{12} + C_{11}}$$
, $\Omega = \frac{(C_{11} - C_{12})}{4 \cdot C_{11} \cdot G_{<110>}}$ [28]

 C_{ik} – the elastic modules are determined by the velocity of transversal $V_{S<100>}$ and longitudinal $V_{L<100>}$ and $V_{L<110>}$ of ultrasonic waves in the crystallographic directions <100> and <110> by the formulas [12]: $C_{11} = \rho \cdot V_{L<100>}^2$, $C_{44} = \rho \cdot V_{S<100>}^2$, $\rho \cdot V_{L<110>}^2 = 0.5 \cdot (C_{11} + C_{12} + 2C_{44})$. As for the lattice parameter *a* and the density of the crystal ρ , their absolute values and temperature course for different ionic crystals are given in [9, 12].

Postesonance range:

$$B = \frac{4 \cdot \Omega \cdot G \cdot b^2 \cdot \Lambda}{\Delta_{\infty} \cdot f_{\infty} \cdot \pi^2} \,. \tag{3}$$

From formulas (1)–(3) it is seen that for performing calculations, the dependences $\Delta_d(f)$ are required, from which parameters Δ_{∞} , f_{∞} , Δ_m , f_m can be determined. Measurement of the dependencies $\Delta_d(f)$ is convenient to carry out by the expanded exponential method [37]. As to the parameter Ω , $G_{<110>}$, and v then they are determined through elastic modules C_{11} , C_{12} , C_{44} , which, in turn, are resolved through experimentally determined velocity of the ultrasonic wave propagation $v_{L<100>}$, $v_{S<100>}$, and $v_{L<110>}$. Given that the dependence of $\Delta_d(f)$ is determined by attenuation α , one can conclude that praactically all the values of formulas (1)-(3) can be determined experimentally by measuring the absorption and velocity of the ultrasound waves in crystals. That is, it is possible to obtain an entire array of experimental data on the same crystal, which is very important. The only parameter to be studied in a separate way is the density of dislocations Λ , which is determined by the method of etch points. In order for the error in determining the parameter Λ to be minimal, the deformation of crystals should be carried out on a small (~ $5 \cdot 10^{-6} \text{ s}^{-1}$) velocity so that the slip bands, which complicate the process of counting the etch points, did not arise [22–25]. In view of the above, it can be stated that the basic methods and technologies on which the emphasis should be placed on the study of the processes of dislocation dynamics under conditions of external influences of different physical nature are the methods of physical acoustics, which allow measuring the speed and absorption of ultrasound in samples, as well as the method of etch points. Of special significance is also the technology of preparation of samples, which directly affects the accuracy of the measurement of physical parameters in formulas (1)–(3).

The purpose of this work is the ground examination of the equipment, which allows us to investigate the features of the process of phonon-dislocation interaction in crystals, to perform thermoactivation analysis of the process of unpinning the dislocations from the stoppers under the influence of temperature and elastic loading, to study the processes of mechanical relaxation in loaded samples [22–25, 27, 29–36].

EXPERIMENTAL TECHNIQUES

In Fig. 1 shows a general view and block scheme of an experimental equipment for precision measurements

of absorption and velocity of ultrasonic waves in crystals.

The specified complex of precision acoustic measurements was used by us for researches of a number of ionic crystals, namely, in NaCl, KCl, KBr, CsI, LiF [22–25, 27, 29–36].



Fig. 1. General view and block scheme of the experimental equipment

The equipment provides the possibility to carry out both high-precision measurements of absolute values of velocity and dislocation attenuation of ultrasound in primary annealed specimens, as well as relative measurements of these parameters, which is important for studying in crystals of processes, as a result of which its structure changes in time (load or relaxation, irradiation of crystals during conducting measurements of acoustic characteristics, heating during measurements, which influences the activation of different processes). The equipment can work both manually and in automatic modes. The automatic mode can be implemented for relative measurements of the acoustic characteristics of the crystal by the selector method.

At the same time, the changes in the attenuation $\Delta \alpha$ and the velocity of passing ultrasonic waves through the sample $\Delta v/v$ caused by the change in the dislocation structure of the crystal are fixed with accuracy not worse than 10^{-4} dB/µs for attenuation and 10^{-4} for the velocity, respectively. The equipment is connected to the tensil machine (see photo in Fig. 1), which enables the recording of the load curve $\sigma(\varepsilon)$ simultaneously with the carrying out of the acoustic measurements. The frequency range of the equipment is 7.5...232.5 MHz, the temperature interval of measurements realized in the works [22-25, 27, 29-36] is 77...450 K.

In Fig. 1 also shows the block diagram of the acoustic device used in this paper and implements speed measurement modes by the selector method and pulse interference method, as well as the attenuation of ultrasound by the expanded exponential method and, if necessary, by the selector method (in the automatic measurement mode). The equipment consists of the generators of rectangular pulses G5-15-1 and 8, the amplifier of video signals 2, the generator of shock excitation of high frequency 3, the amplifier — modulator 9, the receiver 4, the exponent block 5, the

summator of signals 6, oscilloscopes 7, 17, the generator G4-18A sinusoidal oscillations 10, electronic frequency meters Ch3-34A – 11 and 15, selector block 12, speed measurement block 13, attenuation measurement block 14, and recorder 16. In addition, in the block diagram shown there is provided a position for the location of the investigated crystal with the piezo converter. Implement the system of ultrasound scanning of the crystal can be both in the mode of reflection – with one piezo converter, and in the mode of passage – with two piezoelectric sensors.

MEASUREMENT OF ATTENUATION OF ULTRASONIC WAVES IN CRYSTALS BY EXPANDED EXPONENTIAL METHOD AND SELECTOR METHOD

It is known [37] that the ultrasound wave propagating through the medium fades out according to the exponential law $I = I_0 \cdot \exp(-\alpha x)$, where I, I_0 is the current and initial value of the amplitude of the ultrasound; α – the absorption of ultrasound, x is the path passed by ultrasound wave in the sedimentary field. If it enter a mechanical pulse through а piezoelectric crystal, it can obtain a sequence of reflected "bottom" signals. All signals of the specified sequence will be delayed relative to the other for the time of double passage through the sample, each of them will be smaller in amplitude than in the previous one $exp(-\alpha x)$ times. If this sequence is applied to a calibrated exponent, it can quickly and accurately determine the attenuation in the crystal. This is the essence of the expanded exponential method [37]. The mentioned method, in the opinion of the authors [37], is more effective, the less the level of attenuation of in the studied crystals. This is ultrasound understandable: in the case of less attenuation, the sequence of "bottom" signals consists of more signals, and, consequently, this sequence can be more accurately described + by a single exponential curve. For ionic crystals, we have an insignificant level α , so one should expect maximum efficiency from the practical implementation of the method.

The schematically shown in Fig. 1, the equipment in the regimen of the expanded exponential method works in this way. The assignment generator 1 of the rectangular impulses G5-15 produces a rectangular pulse (amplitude 20...50 V), which activates the amplifier of the video signal 2. In addition to the video signal going to 2, the generator 1 also produces a pulse of synchronization that enters the exponents block 5. Block 5 in turn is connected to the 7 - oscilloscope C1-64A through two connectors: the first one feeds the synchroimpulse, which starts the oscilloscope, and the second – to the oscilloscope, the calibrated exponent is directed by block 5. At the generator 1, besides the amplitude and the rectangular shape pulse the delay time of the pulse probing crystal and the clock pulse $(10...50 \,\mu s)$ is set. It gives the possibility starting the oscilloscope twice – a probing impulse (visualizing the sequence of exponential signals on the oscilloscope's screen) and the sync pulse. It let to visualize on the screen of the oscilloscope a calibrated exponent. The distance between the fronts of two rectangular pulses, which is given by the generator 1, is approximately t = 1/f = 2 ms (at a frequency of f = 500 Hz). The duration of rectangular pulses is $\approx 1...2$ µs. In the amplifier of video signals (modulator) 2, the primary signal is substantially amplified (0.5...1 kV) and goes to the generator of shock excitation of high frequency 3, which produces radio pulses (sine wave packets) with a frequency of 7.5...232.5 MHz. Radio signals with a certain frequenc filling of the specified frequency range are sent to a piezoelectric sensor, which is connected to the investigated crystal through a special transition layer. Electric signal, after transformation into a mechanical, acoustic on, moves along the crystal. The sequence of reflected signals through the same piezo sensor is transformed into an electrical signal and enters the receiver 4. Since the "bottom" signals after the passage through the crystal and transformations are too weak (sensor is transmitted with a signal amplitude 300...400 V, and removed 10...20 µV) receiver function, in the first place, is to amplify the signal (at least up to 1 V), and in addition, in detecting the radio signal and its transformation into a video signal.

After passing the signal follower 6, the entire sequence of "bottom" signals goes to the oscilloscope screen. In addition, the rectangular probing signal, falling on the exponential unit 5, after passing RCchains and detecting, is transformed into an exponential, which is observed on the screen along with the sequence of "bottom" signals. Using a calibrated exponent allows to quickly and accurately to determine the attenuation in the samples under study. The photo of the exponential block and real images of probing the investigated crystals of signals made during measurements (Fig. 2) give an idea of the nature of the task being performed. Particular attention should be paid to the correct exponential form of signal amplitude decay. So In the paper [37] it is noted that such a picture is a testimony to the diligence of a number of pre-executed preselected samples of operations (grinding, polishing,

controlling the plane parallelism and mirroring of working surfaces, reliability of contact in the "piezoelectric converter – crystal" system).



Fig. 2. Measurement of the attenuation of ultrasound waves in samples: an exponential block and a demonstration of an expande of a calibrated exponent on the sequence of "bottom" signals

The expanded exponential method allows to measure the attenuation of ultrasound in samples with an accuracy of not less than 5% [37].

The equipment can also work in an automatic mode, which may be convenient in *in situ* studies of external influence the dislocation structure of the crystal. Here, not absolute measurements of the attenuation as such are becoming more important, as its changes are influenced by external factors. In this case, the block scheme of the equipment includes special units that implement the specified measurement mode - an automatic selective attenuation measurement method. Here is a block scheme in Fig.1 works on such algorithm. As before, the signals from the 1 – generator of right angular pulses G5-15 are fed to the video amplifier 2 and then go to the high frequency shock excitation generator 3. After the system of "piezoconverter – crystal – piezo-converter" signals are suitable for the receiver 4, where they are amplified, detected and converted into video signals. At the same time, the sync pulse, after connecting to the circuit through the keys of the corresponding channels, supplies, on the one hand, the selector unit 12, and, on the other hand, triggers of the oscilloscope 17. After opening the synchronization pulse of the selector unit 12, signals from the receiver 4 are received by block 12. The task of block 12 is the formation of a special rectangular selector signal whose position can be changed along the oscillograph scan. It is important for measuring the speed of ultrasound by the selector method, and the amplitude, which is necessary to track the changes in attenuation of ultrasound in the samples. When studying changes in the attenuation of ultrasonic waves in crystals, units 14 and 16 come into operation the attenuation measurement block and the recorder, respectively. From the sequence of "bottom" signals, one chooses one, followed by observations in the course of structural changes in the crystal. This signal gives a selector signal and fixes the output amplitude of the signal. Subsequently, when the amplitude of the signal is changed (for example, due to irradiation of the crystal by X-rays), the amplitude compensation of the signal to the primary level is fixed by the recorder.

MEASUREMENT OF THE VELOCITY OF ULTRASONIC WAVES BY THE SELECTOR METHOD AND THE METHOD OF PULSED INTERFERENCE

If it is necessary to measure the speed of ultrasound with the selectivity method, signals from 12 up to the block of measurement of speed 13. By shifting the rectangular selector signal along the scan line of the oscilloscope and leading it to a signal from the sequence of "bottom" signals, it is possible, for using the frequency meter 15 to determine the delay time between n and n-l signals. Measurement of the state between the working surfaces of the crystal (independently) makes it possible to determine the distance in the double passage of the sample signal, which, at a known time (measured with blocks 13 and 15), enables it to calculate the speed of the ultrasound in the samples under study. The equipment of the ultrasound speed selector method is demonstrated in Fig. 3. The guidance of the selective signal (photo 1) on the "bottom" signals (photo 2-4) makes it possible to determine the time of double passage of sample signals.



Fig. 3. Operation of the equipment in the mode of pulses selection

As already noted, changes in acoustic parameters are recorded with an accuracy of $10^{-4} \text{ dB/}\mu\text{s}$ for measurement of $\Delta \alpha$ and 10^{-4} for measurements of $\Delta v/v$.

Unlike the methods discussed above, the method of pulse interference is based on the consideration of not one but two sequences of echo signals that passed through the sample to be studied. If these two sequence of signals delay one relative to the second at the time of the double passage of the pulse through the sample, then the second reflected signal of the first sequence of echoes will be combined with the first reflected signal of the second sequence. In this case, an interference pattern will be observed on the oscilloscope's screen. The interference minimum – the mutually suppressing of the signals of the two series - will occur under conditions where the signals, equal in duration and amplitude, will be in antiphase. Because the frequency of compensation of the signals f of two sequences is inversely proportional to the time of the double passage of the pulse through the sample, it is possible (with a

known sample length) to determine the velocity of propagation of the ultrasonic wave in the sample:

$$v_{u.s} = \frac{2 \cdot \ell_s}{t_{2\ell}} = 2 \cdot \ell_s \cdot f_k \,.$$

In fact, when measuring the speed with this method, the experimental equipment implements the scheme of the classical interferometer, which is actively used in applied optics (in particular, very widely – in special medical equipment for the diagnosis of damage to internal organs) and provide unique opportunities for highly accurate studies of surfaces of physical objects and internal layers of heterogeneous physical bodies. In our case, an interferometer is acoustic. As with all optical methods related to the effect of interference, are considered the most precise, the same situation in acoustics – the method of pulse interference is the most precise method of physical acoustics and provides the accuracy of determining the absolute value of speed to the fourth digit after the bout and relative error 10^{-5} .

The mode of measurement of ultrasonic velocity by the method of pulsed interference is implemented in the following manner. The rectangular pulse generator 1 produces two signals. Sync pulse signal, as in the case of measuring the attenuation of ultrasound in crystals, triggers the oscilloscope 7 (through the exponential unit 5, which in this case is used simply as a link of the electrical circuit through which the signal is transmitted). Another signal, firstly, is fed to the modulator-amplifier 9, and secondly, it starts the block 8 – the second analogous generator G5-15, which sends a second probing impulse to block 9 with a certain delay. To block 9, in addition to signals from blocks 1 and 8, on the other hand, continuous sinusoidal oscillations from the block 10 - generator of sinusoidal oscillations G4-18A are received. These oscillations can enter 9 only in the time when it is triggered by a signal (amplitude of ~ 30...40 V) from the generator 1 or 8. Thus, from one source of oscillation, two sources are produced, which propagate in the volume of the test sample and interact one to another provide an interference pattern. If we allow ourselves to come up with an associative analogy with optical interferometers, then the amplifier-modulator 9 plays the role of a kind of distributive prism, which makes two of the primary beam of rays. In the future, after passing the system of "piezoelectric converter - crystal - piezoelectric converter", the signals of two sequences come to the receiver 4, amplified, detected, converted to video signals and sent to the oscilloscope 7.

The compensation frequency of the signals is displayed by the generator 10 and fixed exactly 11 - frequency tester Ch3-34 A. Since we are dealing with the interference effect of acoustic waves, it is clear that the compensation frequency will not be one, but a few – a number of values (the usual periodic alternation of the extremums of the interstellar picture). So, at one time, the task of correct processing of experimental data (which was provided ambiguously – by a series of decisions) was determined.

For the processing of the results of the acoustic experiment in the framework of the method of pulsed

interference, the well-known original technique is used [38].

In the series of papers [22–25, 27, 29–36], the method of pulse interference was used episodically for the current control of ultrasound measurements by the selector method and, moreover, all calculations of dynamic and dislocations parameters in the framework of the Granato-Lucke theory were performed on the basis of the data array on the velocity distribution of transverse $V_{S<100>}$ and longitudinal $V_{L<110>}$ ultrasonic wave obtained by the specified method.

Since the successful implementation of the methods of internal friction significantly depends on the degree of pre-preparation to the dimensions of the samples under study (parallelism of the working faces of the crystal, contact in the piezo-converter crystal, metallization of the working surfaces, etc.), then consider the specified technological operations.

PREPARATION OF SAMPLES FOR AN ACOUSTIC EXPERIMENT. CALCULATION OF DIFFRACTION ENERGY LOSSES

The dimensions of the samples under investigation should be in accordance with the recommendations of the authors [37, 39]: the apparent cross section of the sample must be not less than the double diameter of the piezoelectric sensor, and the length should be not less than length of impulse. The authors' request [39] is natural - the size of the tool, which probes the object, must be smaller than the size of this object. As to the labor requirement [37], it is a condition for excluding the error in acoustic measurement, which arises due to the interaction of the sound wave with the "walls" of the medium - the side (not working) surfaces of the crystal. The choice of the size of the future sample in some way causes it to have diffraction losses [37], which are the source of the parasitic signal, adding an error to the measured acoustic parameter. Under diffraction losses, the authors [37] understand the losses due to the output of the sound wave from a cylindrical column, which (theoretically) must limit the trajectory of its motion. The authors of the paper [37] note that, starting at a certain distance from the sample, a sound wave equal in

magnitude $\frac{r^2}{\lambda}$ begins the output of the wave front

beyond the boundary of the cylindrical region (and this is the authors [37] associated with the fact that the piezoelectric converter has the final dimensions), and, therefore, we must take into account the presence of diffraction losses in the measured absorption (which should be deducted from the total measured absorption value). Using the radius of the piezoelectric transducer used by us (r = 4 mm) and the typical frequency values 7.5...232.5 MHz, we performed simple assessments and made sure that, when using the lengths of the sample ~ 30 mm, diffraction loss can only manifest itself frequency channel is 7.5 MHz, and at higher frequencies they are not.

By the way, the disappearance of diffraction losses at high frequencies was demonstrated by the authors [12]. They analyzed the oscillograms of echo signals, taken from the same sample at different frequencies. They istablished that the imperfections of the sequence of echoes (deviation from exponential form) that occurred due to diffraction losses at a frequency of 7.5 MHz, became completely unnoticed in the transition to higher frequencies. Regarding the necessity to account for diffraction losses at the measurements at the frequency of 7.5 MHz, it is always necessary to carefully control the possibility of their presence.

ULTRASONIC LOSSES DUE TO THE NONPARALLELISM OF THE WORKING SURFACES OF THE SAMPLES

The authors [37] provided detailed guidance on the adherence to the accuracy class when processing the working surfaces of the samples. It is indicated that the piezoelectric transducer is sensitive to the phase of the wave falling on it, and in the case of a significant deviation from the right angle when the sound wave propagates in the sample, the reaction of the piezoelectric sensor to the reflected wave changes, and, consequently, the value of the electric signal changes comes to the receiver of the acoustic equipment. At the same time, the losses in the measured ultrasound attenuation are estimated by the formula:

$$\alpha_{u} = \frac{8,68 \cdot \pi^{2} \cdot f^{2} \cdot r^{2} \cdot N \cdot \delta^{2}}{v_{L\langle 100 \rangle} \cdot \ell}$$

where N is the number of reflections of the ultrasoound wave in the sample, δ is the angle of the divergence of the working surfaces. As can be seen from the above formula, the indicated effect increases with the transition to high frequencies. For a qualitative picture of echo-signals in the sample the authors [37] recommend to follow the value of $\delta \sim 5 \cdot 10^{-6}$ rad. Our estimates indicate that in the deviation from the planar parallel of the working faces of the crystal at $\approx 1 \,\mu\text{m/cm}$, the value of δ is $1.7 \cdot 10^{-6}$ rad, which is perfectly acceptable and is in good agreement with the requirements [37]. With such processing of the working surfaces of the crystal, the error in the measured attenuation does not exceed the measurement error of the attenuation by the exponential method and does not affect the accuracy of measurements within this method.

The transition layer between the piezoelectric sensor and the crystal must be made in accordance with the general recommendations [37]. When working in the temperature range of 77...300 K, the lubricant GKZH-94 is a good option, and when transiting to high temperatures of 300...450 K, it is necessary to use VKZH-94 for contact. The technology of creating a contact layer (application of lubricant) is as important for obtaining echo-signals of exponential shape [37] as the high plane parallelism of the working surfaces of the sample.

It should be noted that the method of amplitudeindependent internal friction is very sensitive to the slightest changes in the structure of the crystal, and, taking into account the possibility of thermoactivating analysis [26], it is a definite analytical tool in the study of the nature of point defects at the dislocations. But in the presence of radiation defects in the crystal [29–35], the method indicated does not allow to identify their nature. In this case, the optical method [40–45] should be used simultaneously with the acoustic one, which substantially expands and complements the possibilities of the basic ultrasonic measurement method.

Simultaneous use of both of these methods for registration and identification of defects of radiation origin in crystals is quite applicable to studies of radiation resistance of structural materials [46, 47] and physical processes in solids under the action of ionizing radiation.

CONCLUSIONS

The block scheme of a multifunctional device, which implements the method of amplitudeindependent internal friction in the frequency range of 7.5...232.5 MHz at temperatures of 77...450 K is described. Eauipment provides a possibility to study the features of the phonon-dislocation interaction process in crystals, perform thermal activated analysis of the process of unpinning the dislocations from the stoppers under the influence of temperature and elastic loading, studying the processes of mechanical relaxation in loaded samples.

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ПРИМЕНЕНИЕ АКУСТИЧЕСКОГО ИМПУЛЬСНОГО ЭХО-МЕТОДА ДЛЯ ИССЛЕДОВАНИЯ ДИНАМИЧЕСКИХ И СТРУКТУРНЫХ ДИСЛОКАЦИОННЫХ ХАРАКТЕРИСТИК КРИСТАЛЛОВ

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Описана многофункциональная импульсная установка, которая позволяет методом амплитуднонезависимого внутреннего трения в частотном интервале 7,5...232,5 МГц исследовать особенности процесса фонон-дислокационного взаимодействия в кристаллах, проводить термоактивационный анализ процесса открепления дислокаций от стопоров под действием температуры и упругого нагружения, изучать процессы сбросообразования и механической релаксации в нагруженных образцах квазиупругой и пластической областях деформаций.

ЗАСТОСУВАННЯ АКУСТИЧНОГО ІМПУЛЬСНОГО ЕХО-МЕТОДУ Для дослідження динамічних і структурних дислокаційних характеристик кристалів

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Описано багатофункціональну імпульсну установку, що дозволяє методом амплітудно-незалежного внутрішнього тертя в частотному інтервалі 7,5...232,5 МГц досліджувати особливості процесу фонондислокаційної взаємодії в кристалах, виконувати термоактиваційний аналіз процесу відкріплення дислокацій від стопорів під дією температури і пружного навантаження, вивчати процеси скидоутворення і механічної релаксації в навантажених зразках у квазіпружній і пластичній областях деформацій.