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Acoustic emission defectoscopy of paratellurite crystals

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The possibilities of using the acoustic emission (AE) method for evaluating the defectivity of TeO_2 paratellurite crystals in the process of their elastic-plastic deformation have been investigated. A series of experiments on compression to fracture of paratellurite crystals samples of $6 \times 6 \times 12$ mm in size with registration of AE pulses in the process of loading has been carried out. It is shown that time dependences of primary AE-parameters do not allow to unambiguously determine the structural-mechanical state and the presence of defects in the studied crystals, and a detailed analysis of acoustic signal parameters is required. The most informative AE parameters indicating the formation of crack-like defects have been identified. To establish the possibility of defects detection, the empirical distribution functions of AE pulse duration registered in the process of crystal loading have been constructed. It is shown that the value of the high-level quantile of the empirical distribution function of AE pulse duration can be used as a criterion parameter for establishing the moment of defect formation in the crystal. Its threshold value indicating the occurrence of irreversible damage has been established.

Keywords: acoustic emission, flaw detection, elastic-plastic deformation, paratellurite, cracks, fracture, crystal.

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1. Introduction

Paratellurite TeO_2 is the main material of modern acousto-optics. Thanks to its high acousto-optical (AO) quality, wide spectral range of transparency, manufacturability, and other advantages, it allows development of efficient and compact optoelectronic devices: modulators, deflectors, tunable spectral filters, etc. [1]. Many of them operate under rather severe conditions (with significant temperature differences, vibrations, etc.), which can lead to internal stresses and cracks in the crystal and, as a consequence, deterioration of parameters of the device and its failure. When developing and manufacturing AO devices, various laboratory methods for control of the quality of crystals are used (conoscopy [2], diffractometry [3], etc.), at the same time, little to no diagnostics is performed in the assembled AO device for the stresses arising in the process of operation. Therefore, an urgent task is to diagnose AO cells made of TeO_2 crystals in the process of operation to determine their operating modes under external impacts.

In this study, it is proposed to inspect such crystals for defects using the AE method based on the phenomenon of elastic wave generation during the formation and development of defects in the material. The AE method is highly sensitive to detecting defects when diagnosing damage in the structure of brittle materials [4]. It is used when monitoring the condition of products made of structural and functional materials to assess the stage of the damage accumulation process and the actual condition. The AE method is used to identify developing damage in products made of metals and alloys [5–8] and composite materials [9–11].

Both absolute and complex AE parameters can be used as the main diagnostic indications of damage. According to the results presented in [12–14], crystals can be assessed for defectiveness using primary AE parameters, such as amplitude (u_m), duration (t_{imp}), activity ($\acute{N}$) and average emission frequency values (N_{imp}/t_{imp}). At the same time, often the analysis of primary AE parameters only does not allow for unambiguously determining their threshold values, indicating the development of critical defects in the structure of the product being monitored. To build up more informative models, the results of which correlate with the process of damage accumulation in crystals, complex AE parameters can be used based on calculations of the distribution functions of amplitude (u_m), energy (E_{imp}), and duration (t_{imp}) of recorded signals. As shown in [15–17], at the moment of formation and development of irreversible damage, a change in the distribution parameters of energy and duration of AE pulses is observed, which makes it possible to obtain the analytical dependences $E_{imp}(t_{imp})$ and $u_m(E_{imp})$, which correlate with the degree of damage to the crystals.

The α-paratellurite crystals investigated in this study feature a strong anisotropy of elastic properties [18,19]. The strongest direction is the [001] direction [18,19]. The elastic properties of paratellurite crystals have a temperature dependence [20]: the crystal remains brittle in the temperature range from room temperature to 723 K and is plastically deformed at a temperature of $T > 0.72T_{mel}$, where T_{mel} being melting point [21]. According to various data, for the brittle state of TeO_2 crystals, the elastic limit in the [001] direction of compression can be as high as 150 MPa, and in the [110] direction it can be 110 MPa [22] or 120 MPa [23]. Ultimate strength of the TeO_2 crystal in the [001] direction can be up to 175 MPa, and in the [110] direction it can be up to 134 MPa.

The main types of defects in the structure of paratellurite crystals are point defects and dislocations. The main point defects in paratellurite crystals are defects in the anion sublattice involving oxygen vacancies [24]. Dislocations in paratellurite are formed in the process of crystal growth due to the presence of thermoelastic stresses caused primarily by technological factors [25]. The presence of dislocations has a significant effect on the attenuation of sound waves and worsens the characteristics of AO devices.

This study is devoted to the experimental investigation of defect formation in TeO_2 crystals using the AE method. The main objective of the study is to identify the most informative AE parameters, which can be used to determine the critical state of the crystal preceding fracture in the process of elastic-plastic deformation. The use of methods of statistical analysis of the data recorded under crystal loading with synchronous recording of AE signals will make it possible to identify the main stages of crystal deformation and determine the threshold values of AE parameters corresponding to the processes of formation and development of irreversible damage.

2. Experimental technique

Paratellurite crystal compression experiments were carried out on an Instron 5982 general-purpose testing machine. A sample of TeO_2 crystal was placed vertically on a 8 mm thick flat steel plate. Compression was applied by the upper punch with a floating hinged base to ensure alignment of the crystal axis with the direction of load application. An image of the crystal during the experiment is shown in Figure 1, *a*. The tests were carried out on defect-free paratellurite crystals shaped as $6 \times 6 \times 12$ mm parallelepiped, preliminary inspected for defects using optical systems (laser conoscopy [2]) and X-ray (diffractometry [3]) control. To compare the results of AE diagnostics, defect-free and defective crystals were studied. Two types of samples were tested, which differed in the orientation of their crystallographic axes (Figure 1, *b*). Type 1 sample was cut in the shape of a parallelepiped with its long side parallel to the [001] axis; type 2 sample was cut with the long side parallel to the [110] axis.

Deformation was carried out by moving the upper traverse of the test machine downward at a rate of 0.01 mm/min. The loading consisted of two stages: 1) preloading — increasing the compressive load to 50 N ($\sigma \approx 1.4$ MPa); 2) main loading — smooth increase in compressive load until sample failure.

The AE pulses arising during the compression of the crystal were recorded using a Vallen AMSY-5 system for collecting and processing acoustic signals. VS150-RIC quasi-resonant converters with a built-in preamplifier with a gain of 34 dB were used as receiving converters connected to the AE monitoring system. The measuring transducer was installed on the waveguide in the form of a steel plate and secured with clamps through a layer of contact lubricant at a distance of 25 mm from the crystal.

Before conducting the crystal compression tests, optimal parameters of the Vallen AMSY-5 measuring equipment were determined. According to [26], the discrimination threshold for AE pulses (u_{th}) is determined by the following condition: $u_{th} \geq u_n + 6$ dB (u_{th} — discrimination threshold of AE pulses, u_n — maximum amplitude of noise signals) and it was amounted to $u_{th} = 34$ dB. To eliminate noise signals arising in the process of friction of the upper punch of the testing machine against the crystal surface, bandwidth of the digital filters was chosen equal to $\Delta f_p = 95{-}850$ kHz. To assess the quality of the acoustic contact between the object under test and the receiving transducer, AE pulses were generated on the surface of the side face of the crystal before each test using a Su-Nielsen simulator (mechanical pencil lead breakage). As a result of preliminary tests, the amplitude of the AE pulses from pencil-lead breakage was $u_m = 99.7{-}99.8$ dB, which indicates a low level of acoustic signal attenuation in the waveguide used.

3. Experimental results

At the initial stage of experimental data processing, an assessment was made of changes in the parameters of the flow of AE pulses recorded during the compression of defect-free crystals until fracture. Figure 2 shows the dependences of amplitude (u_m) and duration (t_{imp}) of AE pulses with a superimposed loading curve $\sigma(\tau)$, where σ is compressive stress, τ is time, for two crystals oriented in the [001] and [110] directions.

As shown in the graphs (a, b) in Figure 2, at the first stage of loading ($\sigma \leq 1.4$ MPa; $P \leq 50$ N) of a defect-free paratellurite crystal with the [001] crystallographic direction, no AE pulses were noted. At the initial stage of the second (main) stage of loading ($P \leq 100$ N;

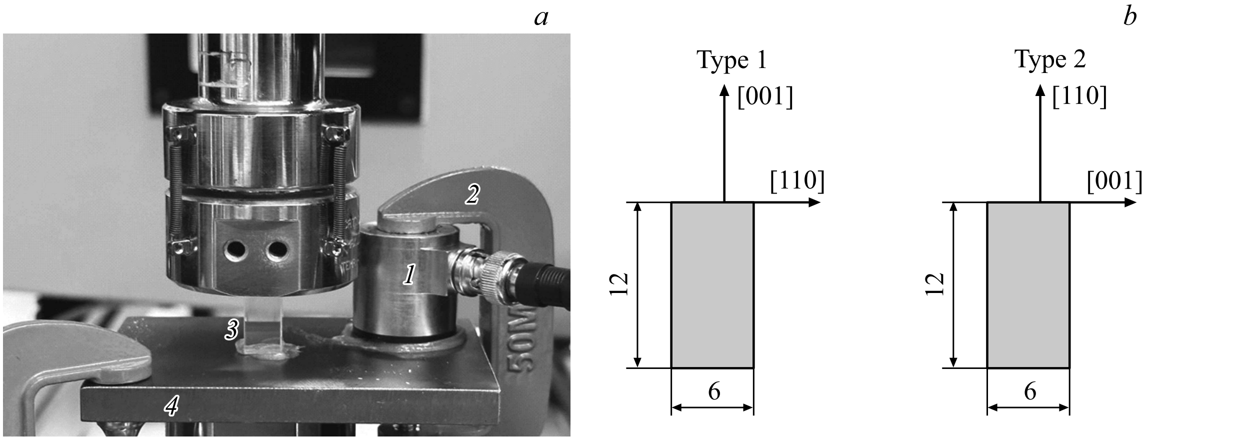

Figure 1. Experimental set-up (a) and diagrams of paratellurite crystal samples (b): *1* — quasi-resonant converter, *2* — clamp, *3* — crystal under study, *4* — waveguide.

$\sigma \leq 2.7$ MPa; $\tau = 110-125$ s) a flow of low-amplitude AE pulses was recorded ($u_m \leq 45$ dB), the duration of which did not exceed $t_{imp} \leq 1000\,\mu$s, which is indicative of the initiation of the process of microdamage accumulation in the material of the monitored product. A further increase in the applied load led to an increase in the amplitude and duration of the recorded signals. At $\tau = 124$ and $\tau = 135$ sec of the loading, local drops of the applied load were recorded from $P = 183$ N ($\sigma = 5.1$ MPa) down to $P = 171$ N ($\sigma = 4.8$ MPa) and from $P = 315$ N ($\sigma = 8.8$ MPa) down to $P = 282$ N ($\sigma = 7.8$ MPa), respectively, which is indicative of the process of relaxation of mechanical stresses during the formation of new surfaces in the course of development of a crack-like defect (Figure $2, a, b$). At the moments of $\tau = 124$ s and $\tau = 135$ s, a local increase in the amplitude and duration of the recorded AE pulses is observed to $u_m = 100$ dB (Figure $2, a$) and $t_{imp} = 60000\,\mu$s (Figure $2, b$), respectively. A local increase in the duration of AE pulses to $t_{imp} = 60000\,\mu$s may be due to the process of friction of the edges of a developing crack [4,27] during compression of a paratellurite single crystal.

The graphs (c, d) in Figure 2 show the amplitude and duration of AE pulses recorded during static compression of a defect-free crystal oriented along the [110] direction. At the initial stage of sample loading, a flow of AE pulses was recorded with their amplitude reaching $u_m = 53$ dB. At the beginning of the second stage of static compression $P \leq 100$ N; $\tau > 110$ s; $\sigma \leq 2.7$ MPa) the appearance of high-amplitude AE pulses is noted, with their duration reaching $12000\,\mu$s.

At the moment of $\tau = 178$ s, pulses with an AE amplitude of $u_m = 100$ dB (Figure $2, c$) and a duration of $t_{imp} = 100000\mu$s were recorded (Figure $2, d$), which is indicative of the occurrence of continuous emission during friction of the edges of the main crack.

From the above-presented results, a general pattern of changes in the primary AE parameters with increasing compressive stress is observed for crystals of both crystallographic directions. When relatively low values of compressive stress are reached ($\sigma \leq 5$ MPa), high-amplitude signals with a long duration appear, indicating the formation of cracks and friction of their edges during loading.

During further processing of the experimental results, a comparison was made of the average values of the primary AE parameters for the defect-free crystals under study. The result of calculating the average values of amplitude (u_m), average emission frequency (N_{imp}/t_{imp}), activity ($\acute{N}$), rise time (t_φ), as well as average values of the local dynamic criterion (m) are presented in the table as numerical characteristics to assess the degree of hazard of the AE sources:

$$m = \frac{\Delta N_\Sigma}{\Delta P}, \quad (1)$$

where ΔN_Σ ia increment of the sum of AE pulses, ΔP is increment of the applied load.

According to the recommendations presented in [26], AE sources are classified into four hazard classes: 1) passive AE source ($m < 1$); 2) active AE source ($m \approx 1$); 3) critically active AE source ($1 < m \leq 6$); 4) catastrophically active AE source ($m > 6$). At the initial stage of loading ($P < 100$ N) of a defect-free sample with the [001] crystallographic direction (see Figure $2, a, b$), the average level of local dynamic criterion was $m = 0.2$. With a further increase in the applied load ($100\,\text{N} \leq P < 200\,\text{N}$; $2.7 \leq \sigma < 5.6$ MPa) there was an increase in the amplitude and activity of recording AE pulses to values of $u_m = 71.2$ dB and $\acute{N} = 18$ imp./s, respectively. It should be noted that significant increases in the primary AE parameters correlated in time with the recorded moment of local drop of the applied load from $P = 183$ N ($\sigma = 5.1$ MPa) down to $P = 171$ N ($\sigma = 4.8$ MPa) (see Figure $2, a, b$), at the same time, the local dynamic criterion did not exceed the value of $m = 1.8$.

When processing a flow of AE pulses recorded during loading of a defect-free sample oriented along the crystallographic direction [110] (see Figure $2, c, d$), the average value of the local dynamic criterion at the $P < 100$ N level

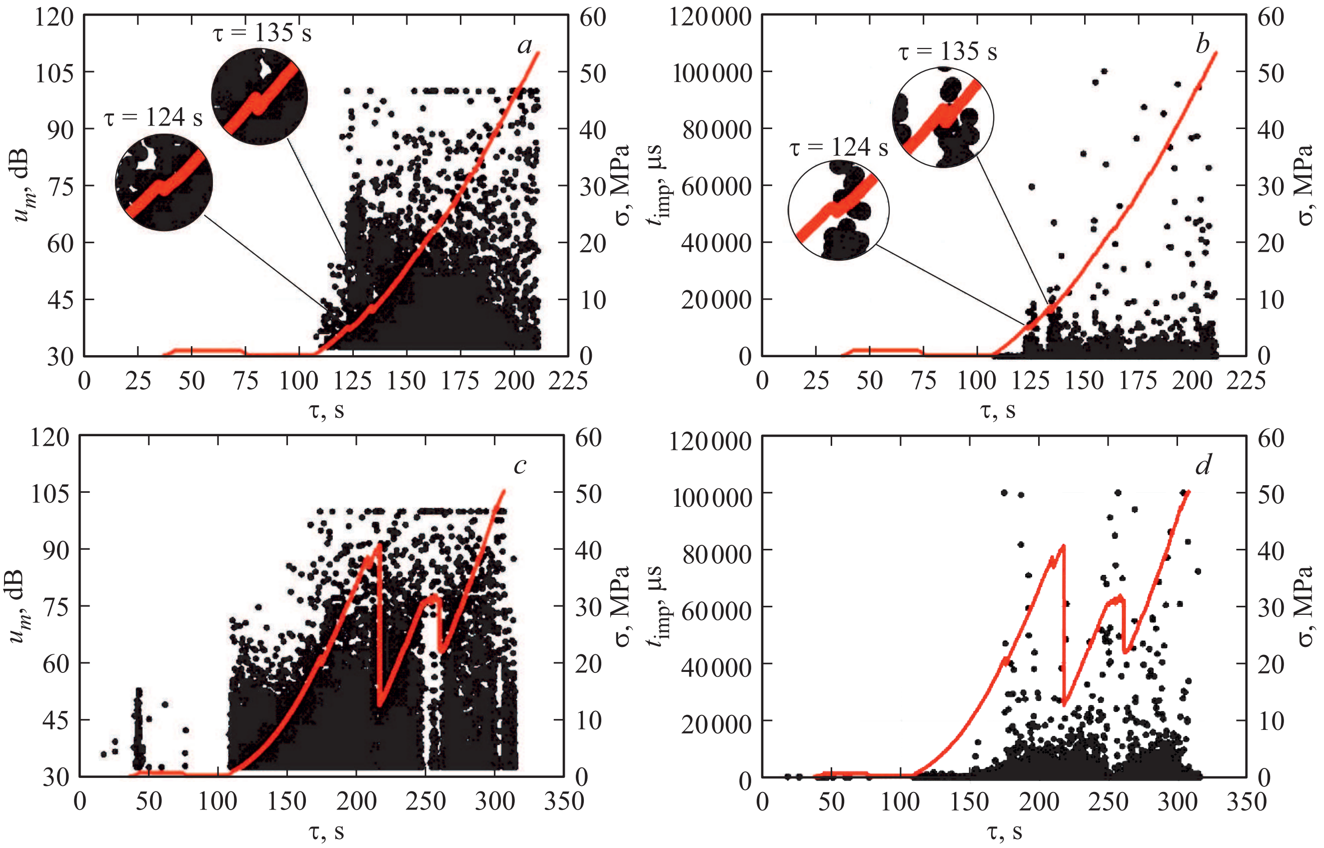

Figure 2. Dependences of amplitude u_m (a, c) and duration t_{imp} (b, d) of AE pulses recorded during compression of defect-free paratellurite crystals along the crystallographic direction [001] (a, b) and [110] (c, d) (red lines show „stress σ — time τ„ loading curves).

of the applied load was as high as $m = 5.3$. Registration of a critically active source of acoustic signals $(m = 5.3)$ at the initial stage of loading does not correlate with the average values of the AE pulse flow parameters $(u_m = 48.2\,\text{dB}$, t_φ=98.1 μs), which makes it difficult to use standard criteria parameters in diagnosing developing damage in brittle materials. A further increase in the applied load led to the appearance of long-duration pulses and the recording of continuous AE.

A similar processing with determination of the primary parameters of acoustic signals recorded during loading was carried out for two defective TeO_2 crystals elongated along the crystallographic directions [001] and [110], respectively (see the table). At the initial stage of loading ($P \leq 200$ N; $\sigma \leq 5.6$ MPa) of the crystal with a defect elongated along the [001] direction, a flow of low-amplitude AE pulses was recorded, the average activity of which was $\acute{N} = 9$ imp./s. In this case, the average duration of the recorded AE pulses was $t_{imp} = 9270\,\mu$s. A further increase in the applied load led to a significant increase in the duration of the recorded AE pulses and the appearance of continuous emission. When loading the crystal with a defect elongated along the [110] direction, a flow of low-amplitude AE pulses was also recorded, the average duration of which reached $t_{imp} = 11540\,\mu$s. A further increase in the applied load led to a significant increase in the duration of the recorded AE pulses and the appearance of continuous emission.

Thus, the analysis of AE data from both defect-free crystals and crystals with microcracks made it possible to establish that for rejecting defective paratellurite crystals, the most informative feature is the relative change in the duration of AE pulses at the applied load level of $P \leq 200$ N ($\sigma \leq 5.6$ MPa).

To increase the reliability of results of the AE diagnostics, an algorithm for estimating changes in the statistical parameters of the flow of recorded AE pulses is proposed. Numerical parameters can be the values of quantiles of empirical distribution functions of the parameter t_{imp}, calculated using a sliding window. Empirical distribution functions for a sample of AE parameters were calculated using a sliding window function F_W^* with a size of W [28]. Value of the F_W^* function was calculated using the following formula:

$$F_W^*(y) = \frac{1}{W}\sum_{i=1}^{W} I(X_i < y), \qquad (2)$$

where W is sample size (window function size); I is number of AE parameters that meet the condition of $X_i < y$; X_i is value of the AE parameter from the sample

Comparison of average values of AE parameters recorded at the main stages of loading of paratellurite crystals

$P_{min} \leq P < P_{max}$, H	u_m, dB	N_{imp}/t_{imp}, kHz	$\acute{N}$, imp./s	t_φ, μs	$m = \Delta N_\Sigma/\Delta P$, imp./N
Crystallographic direction [001] — defect-free sample					
$0 \leq P < 100$	43.1	61.1	1.6	143.9	0.2
$100 \leq P < 200$	71.2	109.9	18	1772.1	1.8
$200 \leq P < 500$	62.6	94.63	48.9	1453.4	3.4
$500 \leq P < 1500$	67.2	100.8	50.6	1278.6	2.7
Crystallographic direction [110] — defect-free sample					
$0 \leq P < 100$	48.2	68.2	31.6	98.1	5.3
$100 \leq P < 200$	47.3	65.4	27.7	87.5	3.3
$200 \leq P < 500$	47.7	74.5	33.9	183.5	2.7
$500 \leq P < 1500$	63.2	96.6	45.5	1131.6	2.8
Crystallographic direction [001] — sample with a defect in the form of a crack					
$0 \leq P < 100$	52.6	86.1	9	164.2	0.9
$100 \leq P < 200$	62.4	120.1	52.2	1215.9	5.5
Crystallographic direction [110] — sample with a defect in the form of a crack					
$0 \leq P < 100$	–	–	–	–	–
$100 \leq P < 200$	83.6	98.8	34.1	381.3	2.1

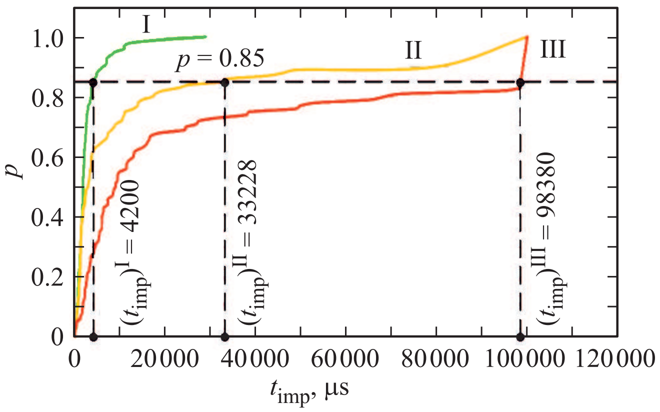

Figure 3. Forms of empirical distribution functions of the parameter t_{imp}, calculated during loading of a defect-free sample oriented in the [001] direction: (I) $P = 130$ H ($\sigma = 3.6$ MPa); (II) $P = 200$ N ($\sigma = 5.6$ MPa); (III) $P = 300$ N ($\sigma = 8.3$ MPa).

$X = (X_1, \ldots, X_i, \ldots, X_W)$; y is threshold value of the AE parameter in the range of $y \in [X_{min}, \ldots, X_{max}]$.

Figure 3 shows as an example forms of the empirical distribution functions of the AE pulse durations calculated using a sliding window of $W = 100$ pulses for one of the defect-free samples under study.

As shown in graph I in Figure 3, at the initial stage of loading of a defect-free sample, the maximum duration of acoustic signals does not exceed $t_{imp} = 29950\,\mu$s. As the applied load increases, the greatest change in the form of the empirical duration distribution function is recorded for quantile values $p > 0.6$. As noted earlier, during the formation and development of the main crack, a flow of AE pulses of long duration is recorded. In this context, the most informative parameter characterizing the appearance of a crack in a crystal is the value of the high-level quantile of the empirical distribution function. Value of the quantile with a level of $p = 0.85$ (see Figure 3) of the empirical distribution function of AE pulse durations $(t_{imp}(p = 0.85))$ was chosen as a numerical criterion correlating with the level of damage to the test object. At the initial stage of loading of the defect-free sample with the crystallographic direction [001] ($P = 130$ N; $\sigma = 3.6$ MPa, curve I in Figure 3), the value of the criterion parameter was $(t_{imp}(p = 0.85)) = 4200\,\mu$s. When the main crack is formed ($P = 200$ N; $\sigma = 5.6$ MPa, curve II in Figure 3), a significant increase in the duration of the recorded AE pulses is observed, which leads to an increase in the quantile to values of $(t_{imp}(p = 0.85)) = 33228\,\mu$s. With a further increase in the compressive load ($P = 300$ N; $\sigma = 8.3$ MPa, curve III in Figure 3), leading to friction of the edges of the developing crack, the appearance of continuous emission is noted, and the quantile value increases to $(t_{imp}(p = 0.85)) = 98380\,\mu$s. Thus, the value of the high-level quantile of the empirical duration distribution function $(t_{imp}(p = 0.85))$ can be used as a criterion parameter for assessing the defectiveness of paratellurite crystals.

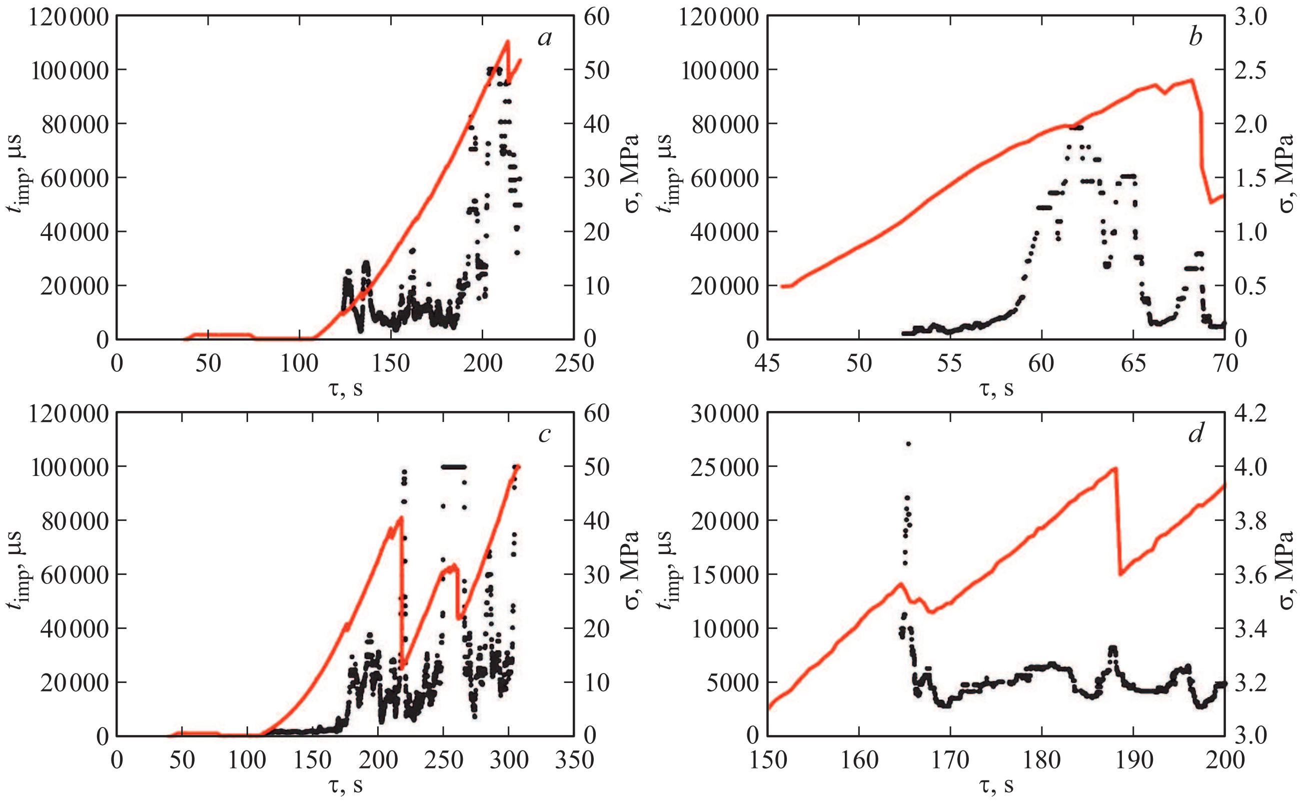

Figure 4. Comparison of the values of the criterion parameter $t_{imp}(p = 0.85)$ calculated in the course of loading of defect-free (a, c) and defective (b, d) samples oriented along the directions of [001] (a, b) and [110] (c, d) (red lines are „voltage σ — time τ„ loading curves).

Figure 4 shows the results of calculating the parameter $(t_{imp}(p = 0.85))$ for all defective and defect-free samples with different crystallographic directions.

As shown in the graph (a) in Figure 4, at $\tau = 124$ and $\tau = 135$ seconds of testing the defect-free sample with the [001] crystallographic direction, moments of local decrease in the applied load were recorded, the occurrence of which correlates with an increase in the criterion parameter to $t_{imp}(p = 0.85) = 27550\,\mu$s and $t_{imp}(p = 0.85) = 29981\,\mu$s, respectively. When the load increases to $P = 500$ N ($\sigma = 13.8$ MPa), the criterion parameter does not exceed $t_{imp}(p = 0.85) = 20000\,\mu$s. At the final stage of crystal loading ($P > 1250$ N; $\sigma = 34.7$ MPa) there is a significant increase in the quantile of the empirical duration distribution function. At the same time, at the moment of fracture ($P = 1985$ N; $\sigma = 55.1$ MPa) of the sample, a continuous flow of acoustic signals was recorded, for which the statistical parameter $t_{imp}(p = 0.85)$ corresponds to $100000\,\mu$s.

When processing the results of AE monitoring of the process of damage accumulation in a defect-free sample elongated in the [110] direction, similar patterns were recorded (Figure 4, c). At the initial stage of crystal loading, the criterion parameter did not exceed $t_{imp}(p = 0.85) = 20000\,\mu$s. At the moment of $\tau = 175$ s, a local decrease in the applied load was recorded from $P = 754$ N down to $P = 711$ N (from $\sigma = 20.9$ MPa down to $\sigma = 19.8$ MPa), the occurrence of which correlates with an increase in the quantile of the duration distribution to the value of $t_{imp}(p = 0.85) = 31200\,\mu$s. At the moment of sample fracture ($P = 1410$ N; $\sigma = 39.2$ MPa), a continuous flow of AE pulses was also recorded, the numerical characteristic of which is an increase in the parameter $t_{imp}(p = 0.85)$ up to $100000\,\mu$s.

As a result of processing the presented experimental data, the authors propose to use as a numerical characteristic of defects in TeO_2 crystal the quantile with a level of $p = 0.85$ of the empirical distribution function of AE pulse durations, not exceeding $t_{imp}(p = 0.85) \leq 20000\,\mu$s.

To verify the proposed algorithm, Figure 4, b shows the values of the criterion parameter for samples with defects. At the initial stage of loading of the defective crystal elongated in the [001] direction to values of ($P \leq 50$ N; $\sigma = 1.4$ MPa), a flow of acoustic signal pulses was recorded with a characteristic increase in the quantile of the empirical function of duration distribution ($t_{imp}(p = 0.85) \leq 10000\,\mu$s). When the load was increased to 75 N ($\sigma = 2.1$ MPa), multiple delaminations were recorded in the structure of the paratellurite crystal, which correlates with an increase in the parameter $t_{imp}(p = 0.85)$ to values of $31000\,\mu$ s.

At the initial stage of loading of the defective crystal with the [110] crystallographic direction (Figure 4, d), no AE pulses were recorded. When the load increases to $P = 128$ N ($\sigma = 3.6$ MPa), there is a sharp increase in the criterion parameter to $t_{imp}(p = 0.85) = 11540\,\mu$s. A further increase in the applied load led to the development of a crack-like defect and a significant increase in the quantile of the empirical duration distribution function ($t_{imp}(p = 0.85) = 27310\,\mu$s).

Thus, the application of the proposed technique made it possible to estimate the defectiveness of paratellurite crystals at low loads based on the values of the empirical distribution function of the duration of AE pulses, not exceeding the threshold value of $t_{imp}(p = 0.85) \leq 20000\,\mu$s.

4. Conclusion

To solve the problem of defect detection of paratellurite crystals considered in this study, a series of experimental investigations were carried out on static compression of crystals until fracture.

Based on the results of plotting the dependences of amplitude (u_m) and duration (t_{imp}), the main features were determined for the changes in the primary AE parameters recorded during static loading of paratellurite crystals in the [001] and [110] crystallographic directions. The general nature of the change in the primary AE parameters with increasing compressive stress for crystals of both crystallographic directions has been established, however, the obtained dependences do not allow for unambiguously identification of the processes of formation and development of irreversible damage in paratellurite crystals.

It is shown that when relatively low compressive stresses are reached ($\sigma \leq 5$ MPa), high-amplitude signals with a long duration appear, indicating the formation of cracks and friction of their edges during loading. It is proposed to use a high-level quantile of the empirical distribution function of AE pulse durations as a criterion parameter to identify defective crystals. A threshold value of the quantile of the empirical duration distribution function of $t_{imp}(p = 0.85) \leq 20000\,\mu$s has been established, the overage of which indicates the development of irreversible damage in the material of the monitored product at low stresses ($\sigma \leq 5$ MPa).

A further development of the proposed method is the development of a calibration characteristic that allows assessing the degree of damage to crystals based on the results of AE diagnostics. To solve this problem, it is proposed to use multifactor analysis, which includes the calculation of not only time parameters but also energy parameters of the flow of recorded signals. In addition, due to the fact that dimensions of the monitored object have a significant impact on the parameters of the acoustic emission signals, to increase the reliability of the AE diagnostics results, it is also necessary to take into account the shape and size of the crystals being monitored.

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Conflict of interest

The authors declare that they have no conflict of interest.

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