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Investigation of local internal stresses under conditions of uniaxial compression of a quartz single crystal

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Based on the analysis of the time intervals between acoustic emission signals obtained in the process of uniaxial compression of a cylindrical sample of a quartz single crystal, local stresses were estimated. It is shown that a decrease in the ability of a material to monotonic stress relaxation with time can serve as a signal for the imminent breakdown of the material.

Keywords: acoustic emission, kinetic concept of strength, local mechanical stresses.

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1. Introduction

Information about the stress fields acting in the volume of the material in the process of deformation is extremely important for understanding the mechanisms that control the development of the destruction process. A significant factor affecting the development of fracture are the local stresses that occur in the given area of the material, which differ significantly from the average ones set by the load. Currently, there are no direct experimental methods to measure the local stresses that occur in the bulk of the material. At the same time, it is these stresses that play the most significant role in the formation of defects and the evolution of the defective structure.

A number of studies [1–5] show that the processes of structural adjustment in quartz at high pressures, heating and phase transformations are accompanied by signals of acoustic emission (AE).

The method of estimating local internal stresses based on the Zhurkov kinetic concept of strength [6,7] and the analysis of the parameters of AE signals [8] was used in this paper. With the help of this method, a study of local stresses arising from uniaxial compression of a cylindrical sample of a quartz single crystal was carried out, and an attempt was made to identify patterns indicating an approximation to a loss of integrity of the sample.

2. Experiment

We used a synthetic quartz crystal of the Z orientation grown by hydrothermal synthesis in the All-Union Research Institute for the Synthesis of Mineral Raw Materials. Cylindrical samples ($d = 10$ mm, $h = 20-24$ mm) were cut from the crystal perpendicular to the facets of the pinacoid 0001, the axis of the sample is the direction $\langle 0001 \rangle$ (Fig. 1).

The samples were subjected to uniaxial quasi-static compression with a loading rate (displacement of loading plates) of $5\,\mu$m/min on an electromechanical machine AGX-Plus (Shimadzu, Japan, maximum force 30 tons). The force was applied parallel to the axis of the cylinder.

A loading was perfomed in several stages, while the maximum force was consistently increased by 4 kN compared to

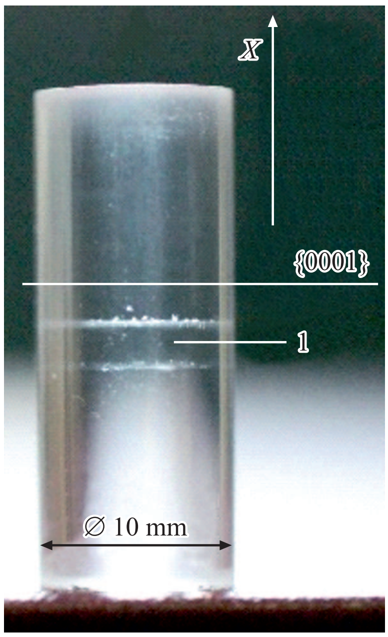

Figure 1. A sample of a quartz single crystal (the number 1 indicates the area of the sample where the seed is located; dotted white dots — the original surfaces of the seed).

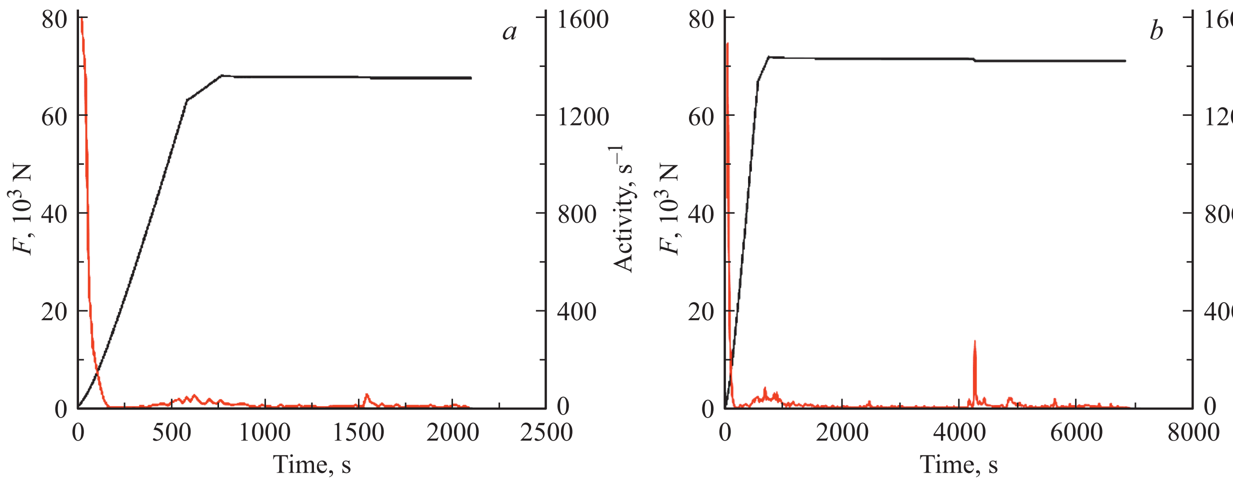

Figure 2. Change of the force applied to the sample (black curve) and acoustic emission activity (red curve) in the first (a) and second (b) loading steps.

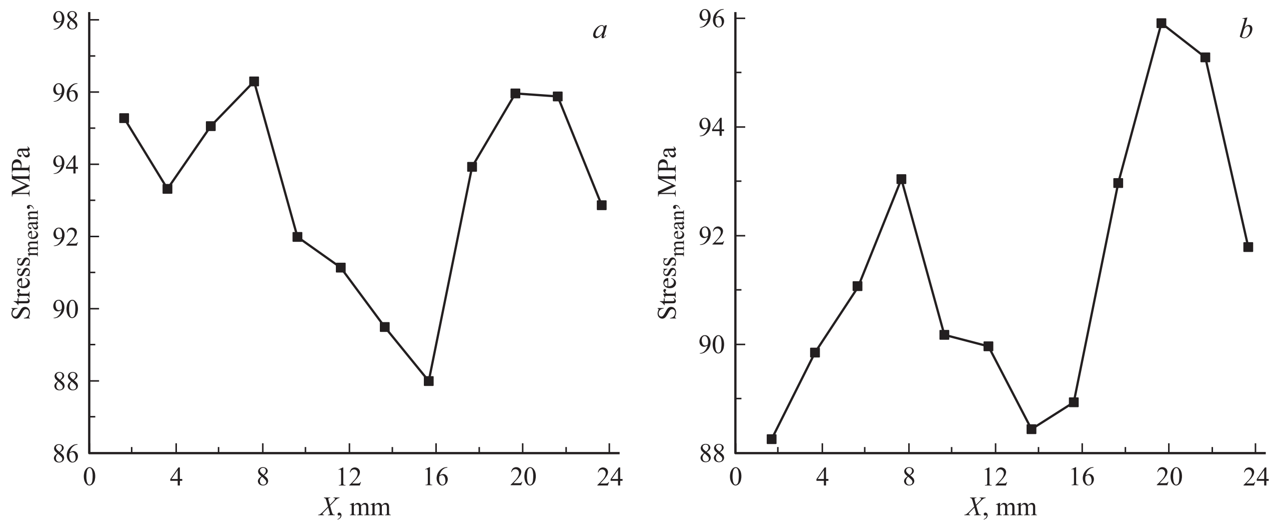

Figure 3. The dependence of the average stresses at the coordinate on the first (a) and second (b) stages for the entire load time.

the previous stage. At each stage, the sample was kept until the activity of AE (the number of signals for a certain period of time, in this case 50 s) did not drop to zero. A total of 18 loading stages were performed. However, the number of AE signals recorded in the first 16 stages (i.e. up to 64 kN) was insufficient for statistical analysis. In this regard, an analysis of the last two loading stages was carried out (hereinafter we will call them „stage 1“ and „stage 2“), on the first of them compression was held up to a force of 68 kN, on the second — to 72 kN (Fig. 2). After the experiment was completed, the sample retained its integrity.

In the course of sample loading, acoustic emission signals were recorded in real time using an Amsy-5 Vallen system (Germany). Two piezoelectric converters AE105A (frequency range of 450–1150 kHz) were attached to the ends of the sample. The accuracy of determining the coordinates of the hypocenters of the AE signals is not worse than 2 mm. Each AE signal was characterized by the radiation time, the coordinate of the source by the height of the sample, and the energy. The experiment details have been described earlier, for instance, [10].

3. Results and discussion

Local compressive stresses were determined (calculated) on the basis of Zhurkov kinetic concept [6,7] and experimental data obtained by acoustic emission.

Detailed studies of the defective structure, conducted using X-ray computed tomography and X-ray diffraction imaging (XDI) [11], showed that even at low loads in a sample of a single crystal of quartz, microcracks are formed, which are the sources of acoustic emission signals.

In agreement with the kinetic concept of S.N. Zhurkov, the external load removes the material from the equilibrium state. As a response to this effect, the material passes into a

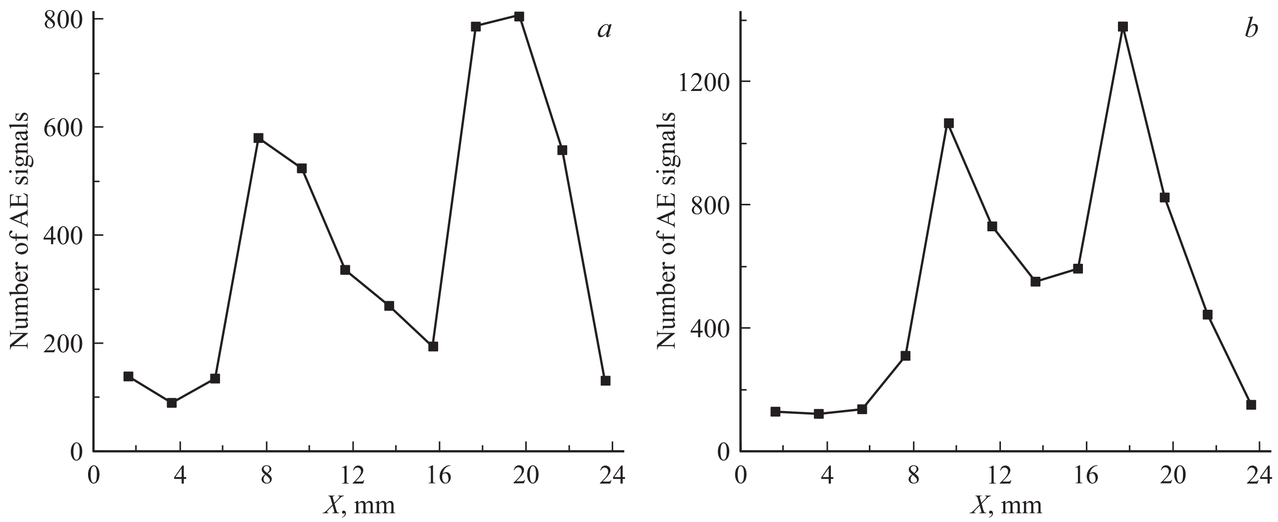

Figure 4. Distribution of the number of AE signals by coordinate (sample height) in the first (a) and second (b) loading steps.

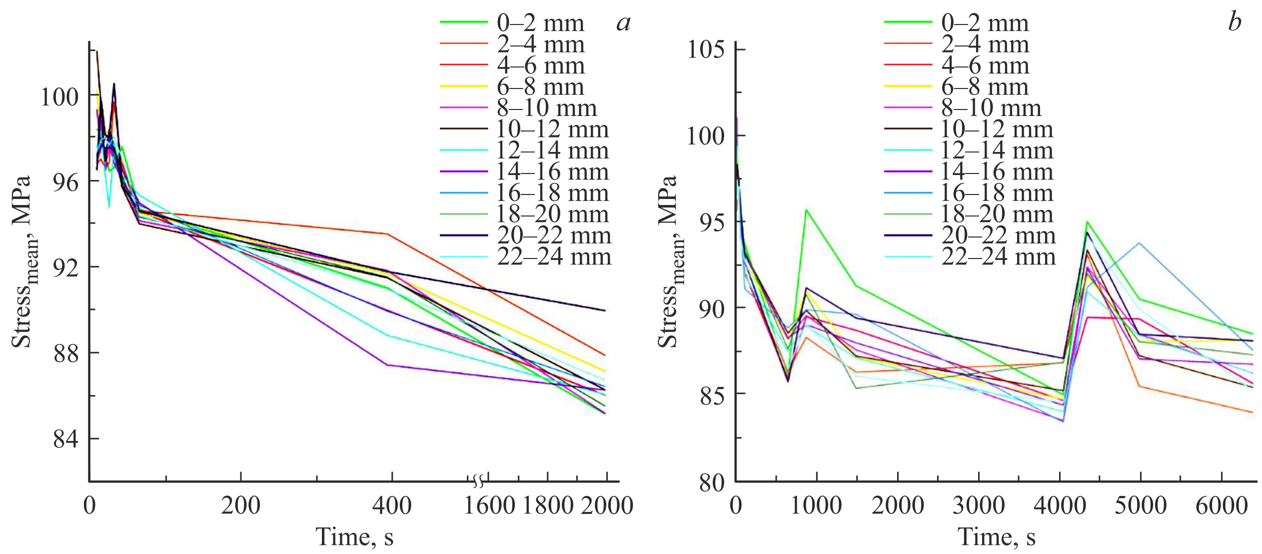

Figure 5. The dependence of local stresses in parts of the sample with a height of 2 mm depending on the time on the first (a) and second (b) loading stages.

new equilibrium state, while passing through a sequence of metastable states with lifetimes Δt_k which can be obtained from acoustic emission data and represent pauses between successive acoustic events.

The stresses themselves are calculated by the formula

$$\sigma(t_k) = \frac{U_0 + kT \ln \frac{\tau_0}{\Delta t_k}}{\gamma}.$$

To analyze the change in local stresses by coordinate, the following procedure was performed: the AE signals were distributed according to the height of the sample into slices with 2 mm height, then the average stresses were calculated in each of this slice. As a result, the dependencies of the average stresses on the coordinate (Fig. 3, a, b) for the entire duration of the experiment were obtained. It can be seen that the maximum local stresses are reached in spatial areas with coordinates ≈ 5 and 18 mm.

Figure 4, a, b shows the distributions of the number of AE signals by the height of the sample at the first (a) and second (b) loading stages. A comparison of the dependencies presented in Figs. 3 and 4 allows us to say that the coordinates of the centers of destruction (~ 5 and 18 mm) correspond to the coordinates of the areas of increased local stresses.

Figure 5, a, b shows the dependencies of local stresses in slices of the sample with a height of 2 mm as a function of time. To construct these dependencies, the time series of parameters of the acoustic emission signals were divided into non-overlapping samples containing 500 signals. Then, for each sequence, a height partition (2 mm) was performed.

Each of the sampling events fell into one of these areas. For each area, the average values of local stresses were calculated.

We have observed a same nature of dependencies earlier [8] in similar experiments on deformation of sandstone.

As follows from Fig. 2, *a, b*, the greatest activity of AE signals (and therefore the greater intensity of cracking) was achieved at the initial moments of time.

In Figure 5, *a, b* it can be seen that the material at the first loading stage has a greater ability to monotonous stress relaxation than at the second.

Based on this observation, the following pattern can be noted: with an increase in the degree of destruction of the material, its ability to monotonous relaxation of stresses decreases.

4. Conclusion

Due to the evolution of the defective structure — the formation and propagation of cracks — the nature (features) of the development of destruction in a sample of a single crystal of quartz acquired similarities with the destruction of sandstone (heterogeneous material).

It is established that in the process of loading a quartz sample, there is a decrease in local stresses. At the same time, with an increase in the degree of destruction of the material, its ability to monotonous relaxation of stresses also decreased.

Conflict of interest

The authors declare that they have no conflict of interest.

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