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Formation of „primary" cracks upon fracture of quartz

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The spectrum was obtained and the time dependences of the fractoluminescence signals were studied during the destruction of the quartz surface by „microcutting" perpendicular to the (0001) axis. An analysis of the obtained data showed that clusters of the 4 smallest „primary" cracks appear during fracture. The formation of cracks is associated with the destruction of barriers that prevent the movement of dislocations along slip planes. The crack sizes are several nm, and the growth rate is several m/s. The size distribution of cracks has a power law form.

Keywords: „primary" nanocracks, fractoluminescence, quartz.

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1. Introduction

Cracks in metals, polymers and crystals have been studied by now with a large number of methods: fractoluminescence (FL), electron microscopy, acoustic emission, X-ray microtomography, etc. [1–15]. It is found that the crystal fracture process begins with the accumulation and consolidation of the smallest „primary" cracks with sizes of a few nm [6–9]. They are formed when dislocations break through barriers that prevent them from moving along sliding planes [6–9,16]. It is found that in a stressed solid, cracks first accumulate independently of each other throughout the volume, and then they merge [1–15], enlarge and form a fracture centre — a main crack. All of these processes start with the formation of „primary" cracks.

In the present work, we investigate the size distribution of primary cracks on the quartz surface at the moment of fracture, when their concentration becomes so high that they begin to interact with each other. In this case, the distribution of „primary" cracks $P(l)$ by size l can be described by a power function [10–13]:

$$P(l) \sim l^{\beta}, \qquad (1)$$

where β is usually ≈ -1 [10–13,17,18].

As a classic example, the Gutenberg–Richter law — the relation between the number of earthquakes and their energy [19]. The same law holds for the distribution of acoustic signals in rock fracture [10–15].

The nature of the power law distribution is related to the strong interdependence of the birth and development of an ensemble of cracks, which leads to their enlargement and avalanche-like fracture of the body [20,21].

2. Research target and methods

The quartz mono-crystal samples were fractured by „micro-cutting" of the surface. The setup layout is shown in Fig. [6,7]. For this purpose, samples were prepared, in the form of parallelepipeds with dimensions $2 \times 2 \times 4$ cm, whose axis (0001) is parallel to their long edge. One end of the parallelepiped was pressed against a steel disc with diamond microcrystals with linear dimensions $\approx 7\,\mu$m glued to its surface. The plate is seated on the axle of the electric motor. Once it was switched on, the disc began to rotate at 5 m/s, the „micro-crystals cut" the surface of the quartz crystal, resulting in FL (photo of the setup shown in jcite6).

The FL spectrum of quartz (Fig. 1) was recorded with an AvaSpec-ULSi2048L-USB2 fiber-optic spectrometer.

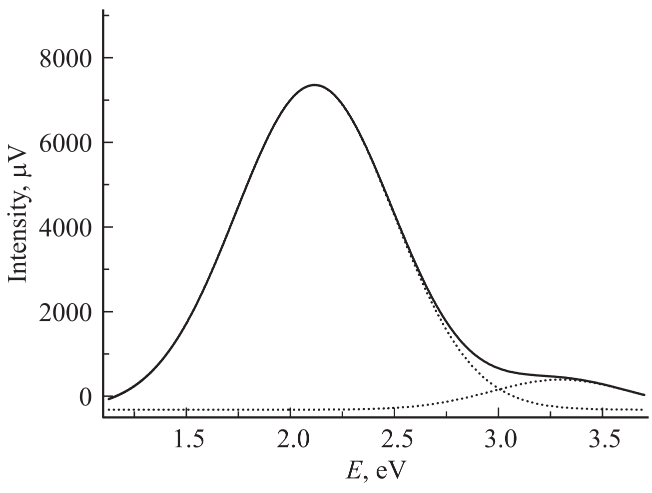

Figure 1. FL spectrum of quartz.

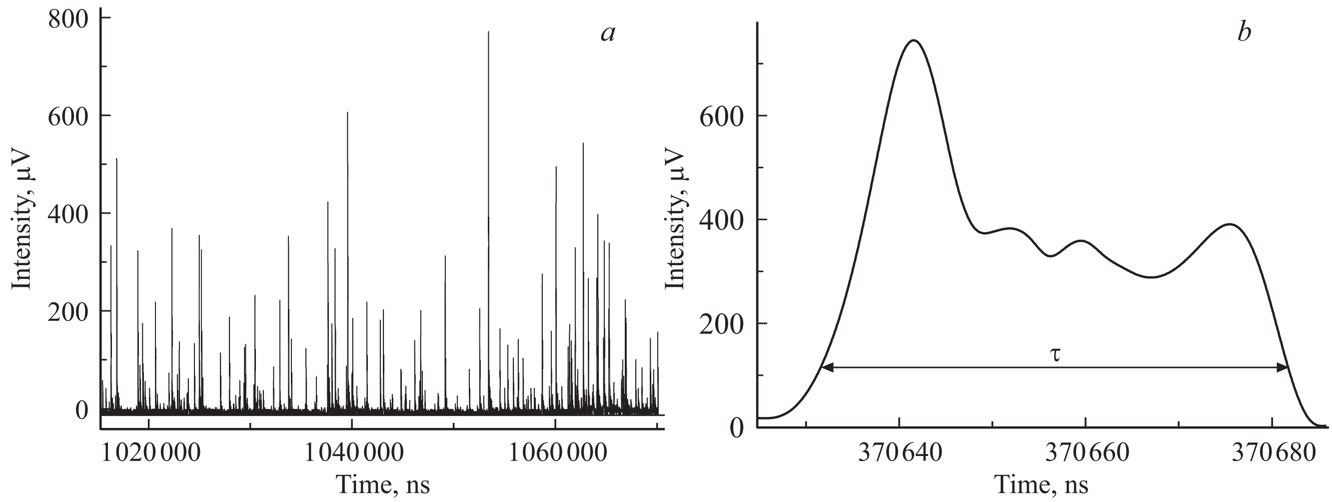

Figure 2. Fragment of the time dependence of the FL (*a*) and single FL (*b*) signals.

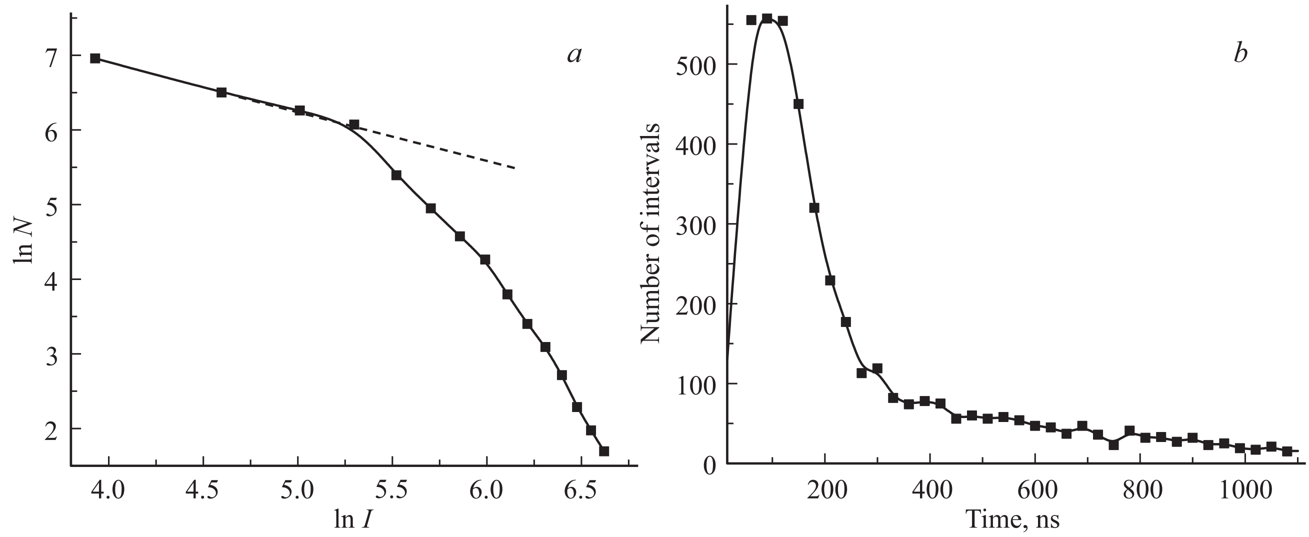

Figure 3. Intensity distributions of the first maximum in the FL (*a*) signals and the intervals between the signals (*b*).

To investigate the time dependence of FL intensity, the radiation was focused onto the surface of a PEM-136 photomultiplier tube. The electrical voltage from its output was fed to the input of an ADC-3112 analog-to-digital converter from ACTACOM. The ADC output voltage was written to the PC memory every 2 ns.

Quartz has a three-dimensional framework of SiO_4 tetrahedrons. Barrier breakthrough and the formation of „primary" cracks in this crystal occurs by breaking $-$Si$-$O$-$Si bonds. The free radicals — $\equiv$ Si$-$O• produced by the ruptures are in an excited electron state. During the transition to the ground state, excitation energy is emitted in the form of radiation in the visible area of the spectrum — FL. By analyzing the appearance and dynamics of FL signal accumulation with a time resolution of 2 ns, barrier breakthroughs and the formation of „primary" fractures can be traced.

3. FL spectrum and dynamics

In the FL spectrum (Fig. 1), 2 bands were observed during quartz fracture: intense — 2.12 and weak — 3.3 eV. The first band corresponds to radicals $\equiv$ Si$-$O•, and the second (3.3 eV) — FL — $[AlO_4/M^+]^0$ centers, where M^+ — Li^+, Na^+, H^+ [22,23], located on the banks of „primary" nano-cracks. These cracks in quartz are formed when the barriers that prevent dislocations from moving along sliding planes break through [6,7]. When „micro-cutting" quartz crystals perpendicular to the axis (0001), the nano-cracks form clusters of 4s, appearing one after another through $\approx 8-15$ ns [6,7]. The appearance of each crack produces a maximum in the FL [24] signal. Therefore, the FL time dependence contains 4 superimposed maxima in each signal (Fig. 2).

A total of 3300 FL signals were analyzed. The duration of each signal $\tau \sim 50$ ns. The intensity of the signals, proportional to the number of cracks, varied by an order of magnitude (Fig. 2). The first maximum is ≈ 2 times the other three maximums in the cluster. It corresponds to the largest cracks in the cluster.

4. Distribution of „primary“ cracks by dimension

Figure 3 shows the intensity distribution I_m of the first maximum in FL signals in double logarithmic coordinates. Up to $I_m \approx 200\,\mu$V it is linear, the tangent of the angle of the straight line ≈ -1. Above $200\,\mu$V the relation becomes non-linear. Under our intensity measurement conditions, its value $I_m = 1\,\mu$V corresponds to ≈ 0.165 nm (the method for estimating crack size from the FL intensity is described in [6]). This shows that the distribution of „primary“ cracks smaller than $l \approx 200 * 0.165 \approx 30$ nm follows a power law.

Another way to test this conclusion — is to find the ratio of the distance between clusters L to their size l. In work [23] it was found, assuming the cracks are in the form of a ball, a value of $L/l \approx 3$.

Let’s turn again to Fig. 3. It follows that the interval between maxima — $T \approx 120$ ns, and the „lifetime“ of the clusters, as noted above $\tau \approx 50$ ns. Then also $T/\tau \approx L/l \approx 2.4$. This result is consistent with the assumption that the size distribution of clusters of primary nano-cracks has a power-law shape.

5. Conclusion

„Micro-cutting“ of the quartz crystal surface produces primary nano-cracks that are — a few nm in size. The surface of the crack banks contain excited $\equiv$ Si-O● radicals, which are formed when the Si$-$O$-$Si bonds of the quartz crystal lattice are broken. When the excitation relaxes, FL signals are generated. The distribution of „primary“ cracks in terms of size is described by a power dependence. This is true for crack sizes smaller than 30 nm.

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Conflict of interest

The authors declare that they have no conflict of interest.

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