Studying internal structure of quartz by broadband ultrasonic tomography under cyclic compression

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Abstract. The article considers the possibility of studying and identifying defects in the internal structure of synthetic quartz crystals using the parameters of acoustic emission and laser-ultrasound tomography. In the course of cyclic compressive loading, the nature of the propagation of discontinuities in a sample was investigated. It is revealed that the properties of the material, the inhomogeneity of which is associated with the presence of microdefects and anisotropy, change in the initial stages of mechanical load. With repeated application of the load on the sample, previously formed defects cause significant changes in its internal structure. With the help of numerical modeling, the obtained results were verified.

1. Introduction
Quartz (SiO₂), the most abundant mineral found in the earth’s crust, has eleven crystalline polymorphs determined by the temperature and pressure of the environment during the time of crystallization [1]. Quartz is also a promising material for electronics and nanophotonic devices and systems [2]. High purity silica glass, with its low loss and large transparency window [3], was the key element that enabled low-loss optical fiber technology and long-distance communications. Silicon dioxide is also a popular material for nonlinear optics [4].

This paper discusses the use of the acoustic emission and laser-ultrasound tomography [5-8] study the development of defects in synthetic quartz crystals under uniaxial mechanical load. The experimental results are compared with numerical simulation data.

2. Materials and methods
2.1. Materials
Uniaxial compression experiments were carried out on structurally homogeneous samples of synthetic quartz (density \( \rho = 2.65 \text{ g/cm}^3 \)) in the form of parallelepipeds with characteristic edge lengths: \( a = 5 \text{ cm} \), \( b = 2.5 \text{ cm} \), \( c = 2.5 \text{ cm} \), the crystals having different elastic properties along the crystallophysical axes due to symmetry. Local heterogeneities in the samples (two-dimensional, one-dimensional, and point defects) were no more than \( 10^{-3} \text{g/cm}^6 \) in size. The physical and mechanical properties of the test material before the testing are given in Table 1.
Table 1. Physical and mechanical properties of test material

| Axis | Longitudinal waves velocity $c_L$ (m/s) | Transverse wave velocity $c_T$ (m/s) | Elastic modulus $E_y$ (GPa) | Poisson ratio $\nu$ | Uniaxial compression strength $\sigma_u$ (MPa) |
|------|--------------------------------------|-------------------------------------|-----------------------------|----------------------|----------------------------------|
| X    | 5750                                 | 3360                                | 74                          | 0.24                 | 300                              |
| Y    | 6010                                 | 4570                                | 90                          | 0.20                 | 315                              |
| Z    | 6320                                 | 4680                                | 104                         | 0.11                 | 350                              |

2.2. Experiment setups

A manual press was used to perform cyclic compression testing of synthetic quartz at a constant speed of 2900 N/s. A special film was placed between the samples and the press plates to prevent friction. The maximum stress was 65 MPa (= 0.2$\sigma_u$) in the first loading cycle and 79 MPa (= 0.27$\sigma_u$) during the second one. Acoustic emission during loading was registered by a piezoelectric ceramic sensor fixed on the sample. The acoustic signal generated by load-induced changes in synthetic quartz was converted into an electric signal to be fed to a computing unit. During the experiment, the number of times the acoustic emission signal exceeds a preset threshold during the observation interval was recorded; this parameter carries information about the cumulative damage to the test object as a result of external action.

The internal structure of rock specimens after second cycle of loading was imaged by laser ultrasonic tomography [5-8]. Optical radiation of a Q-switched Nd:YAG laser (wavelength $\lambda = 1064$ nm, pulse duration $\tau = 10$ ns, pulse rate 20 Hz) is delivered by a multimode silica fiber (NA 0.22) to an optoacoustic generator for laser excitation of ultrasonic pulse. The optical beam is spread on the flat surface of the generator by a system of lenses. A flat-concave acoustical lens focuses the laser ultrasonic probing pulse on the sample. It passes through immersion liquid (water), partially reflects from the surface of the sample under inspection, and passes backwards through the same lens to be recorded by a plane piezoelectric detector array of 16 polyvinylidene fluoride transducers (length, 20 mm; width, 1 mm; gap between transducers, 1 mm). Detector array provides effective reception of ultrasound in the frequency band of 1.6–9 MHz. Although the transducers are flat, they are glued to the flat surface of the acoustic lens. This makes the array effectively cylindrical (focal distance, 40.1 mm). To obtain a high-resolution tomogram, the sample has to be moved to the array focus by the three translational axes of the four-coordinate positioning system. The fourth axis is rotational, and it is designed for 3D step-by-step scanning of the samples.

2.3. Basic computer modeling principles

The experimental curves were compared with the results of numerical simulation of the behavior of synthetic quartz under uniaxial compression. The numerical simulation was performed using Abaqus 6.13 Software whose Abaqus/Explicit module considers the sample to be firmly fixed between the lower fixed plate and the upper movable one of the compression testing press. The plates were regarded as discrete rigid elements having the same size. Increments in the load applied to the upper plate to move the plate along the Y axis were simulated by an increase in the initial displacement of the plate at each time point. The numerical simulation was based on the Drucker-Prager model that describes the behavior of material under load. The model takes the strength characteristics into account, viz. specific weight $\gamma = 26.5 \text{kN/m}^3$, elasticity modulus $E = 100 \text{GPa}$, angle of internal friction $\phi = 31^\circ$, angle of dilatancy $\psi = 20^\circ$, and compression strength $\sigma_u = 300 \text{ MPa}$.

3. Results and conclusions

After two consecutive cycles of compressive loading, synthetic quartz was investigated using a laser ultrasonic tomography. The tomography system can produce images of multiple microcracks formed...
mainly along the axis in any cross-section perpendicular to one of the test sample’s seven unloaded edges, when scanning along the line of intersection between an edge and the cross-section perpendicular to the edge. Cross section of middle region with microcracks after second cycle of loading are shown in Fig. 1. The darker narrow elongated areas with rough boundaries (corresponding to phase changes of the acoustic signal in reflection) are the images of microcracks formed after two consecutive loading cycles.

**Figure 1.** Cross section of middle region with microcracks after second cycle of loading.

The study has shown that two consecutive cycles of loading cause structural defects 3-8 mm in size throughout the synthetic quartz sample, not just near the plane \( Y=0 \) contacting with the press plate. In the central region of the sample cracks are still longer. They are mainly located near the \( Y \) axis, and their length reaches 15 mm.

To verify the experimental results, numerical simulation of the behavior of a synthetic quartz crystal under uniaxial compression along the axis was performed. Fig. 2 shows the results of numerical simulation of the equivalent Mises stress \( \sigma_M = \left[ \left( \sigma_1 - \sigma_2 \right)^2 + \left( \sigma_2 - \sigma_3 \right)^2 + \left( \sigma_3 - \sigma_1 \right)^2 \right]^{1/2} \) obtained on the base of the Drucker-Prager model. In last formula \( \sigma_1 \), \( \sigma_2 \) and \( \sigma_3 \) are three principal normal stresses correspondingly ( \( \sigma_1 > \sigma_2 > \sigma_3 \) ). In the central part of the side edges and near the plane \( Y = 0 \), there are small areas with \( \sigma_M \) three times greater than the normal stresses in other parts of the sample. Areas with the highest \( \sigma_M \) are situated in the same sample parts.

**Figure 2.** The simulated distribution of Mises stresses \( \sigma_M \) across the sample’s faces parallel to the \( Y \) axis at an external load of 75 MPa.
The behavior of a synthetic quartz crystal whose properties are similar to those of natural rocks has been investigated under cyclic loading with the aid of acoustic emission and laser ultrasonic tomography using an automated laser ultrasound tomography system. Two consecutive cycles of loading were performed, the longest edge of the crystal being oriented along the $Y$ crystallophysical axis. The experimental results were interpreted with the help of the numerical solution of the problem for a sample under uniaxial compression; the properties of the sample were described using the Drucker-Prager model, which takes the strength characteristics into account. The properties of the material, the heterogeneity of which is related to the presence of a small number of micro-defects and anisotropy, change at the early stages of mechanical loading. During repeated loading, earlier formed defects cause significant changes in the structure. After two consecutive loading cycles, defects up to 15 mm in size occur throughout the synthetic quartz crystal.

References

[1] Friedt J M and Carry E 2007 Introduction to the quartz tuning fork *American Journal of Physics* 75 pp 415–422
[2] Li G, Winick K A, Said A A, Dugan M and Bado P 2006 Waveguide electro-optic modulator in fused silica fabricated by femtosecond laser direct writing and thermal poling *Optic Letters* 31 p 739
[3] Ilchenko S, Savchenkov A A, Byrd J, Solomatine I, Matsko A B, Seidel D and Maleki L 2008 Crystal quartz optical whispering-gallery resonators *Optic Letters* 33 pp 1569–1571
[4] Brinker C J and Clem P G 2013 Quartz on silicon *Science* 340 pp 818–819
[5] Shibaev I A, Cherepetskaya E B, Bychkov A S, Zarubin V P and Ivanov P N 2018 Evaluation of the internal structure of dolerite specimens using X-ray and laser ultrasonic tomography *International Journal of Civil Engineering and Technology* 9 pp 84-92
[6] Cherepetskaya E B, Karabutov A A, Makarov V A, Mironova E A, Shibaev I A, Vysotin N G and Morozov D V 2017 Internal Structure Research of Shungite by Broadband Ultrasonic Spectroscopy *Key Engineering Materials* 755 pp 242-247
[7] Kravcov A, Svoboda P, Konvalinka A, Cherepetskaya E B, Karabutov A, Morozov D V and Shibaev I A 2017 Laser-Ultrasonic Testing of the Structure and Properties of Concrete and Carbon Fiber-Reinforced Plastics *Key Engineering Materials* 722 pp 267-272
[8] Kravcov A, Shibaev I A, Blokhin D I, Bychkov A S, Cherepetskaya E B, Krapivnoi M M and Zarubin V 2018 Examination of structural members of aerial vehicles by laser ultrasonic *International Journal of Civil Engineering and Technology* 11 pp 2258-2265

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