STRUCTURAL TRANSFORMATIONS UNDER HIGH-PRESSURE FAILURE OF SANDSTONE SAMPLES

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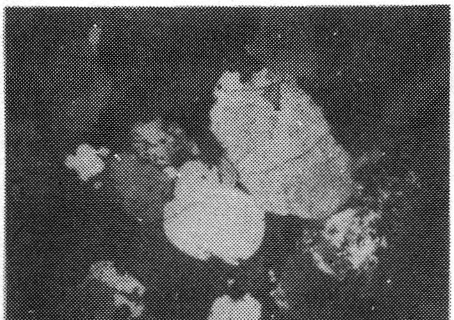
At the plant of nonuniform three-axes compression (TACP) [1] designed by engineers of Donetsk Phys.-Techn. Inst. of Ukr. Acad. Sci., we carried out experimental studies of destruction of prizma-shaped sandstone samples at different loading schemes. The plant permits to produce stresses along three axes up to 700 MPa.

It was found that destruction of samples in the nonuniform field of compressing stresses (e.g. when $\sigma_1 = 150$ MPa, $\sigma_2 = 20$ MPa, and $\sigma_3 = 0$) propagation of cracks is mainly along quartz-grain boundaries. In Fig. 1 it is seen that grains are very densely situated. The path of cracks in the circumvention of them is hampered. One can see grains with mozaic reduction, edges of most grains are splintered. At the growth of σ_3-stress the splinter-evoking stresses σ_1 and σ_2 are also increased.

At deformation of sandstone samples in the volume field of compressing stresses one observes two (of basic three) mechanizms of their destruction: cataclase with splintering, granulation and grain adjustment, at which the main role is played by both friction between grains and siliding with twinning in calcites and feldspars with insignificant friction [2]. The third basic mechanism is recrystallization in the process of local melting or hard diffusion. Recrystallization and phase transformations might take place along quartz-grain boundaries and along interfaces of shear as a result of so-called chip-melting in the narrow localized zones. Brittle chipping as a result of shear occurs rapidly. One can ignore heat losses by heat conduction during this process. According to [3] heat generated per unit of area of plane fracture (considering that shear stresses in the shearing plane are 80–100 MPa and the distance of shear is several tenths of a millimeter) is ca. 1.9 kcal. Since to melt Iqu.cm of dry silica one needs ca. 1 kcal, it is highly probable that shear destruction involves melting.



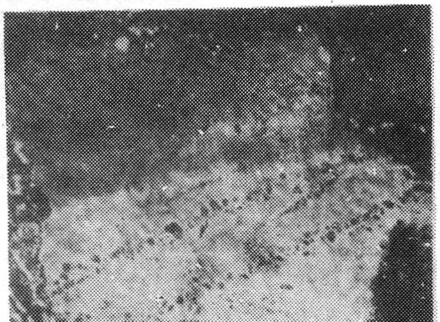


Fig. 1. Growth of cracks in sandstone samples over quartz grains

Fig. 2. Lines of sliding, quartz grains

Observation of grain edges and crack sides with the aid of polarized microscopy witness that the values of diffraction coefficients of grain edges at the interface of grain boundaries and chipping cracks are higher as compared with diffraction coefficient values of bulk of grains. The relative diffraction coefficients were estimated by observing the shift of Beckk's strip [4] (at raising of the microscope drawtube the strip is shifted to less diffracted mineral and vice versa). It was found that the strip is shifted at raising of the microscope drawtube from the grain edge to the bulk of grains. Thus, our conclusion is confirmed. Besides, edges of quartz grains are always yellow, which according to the Lodochnikov dispersion effect indicates that their diffraction coefficients are lower. The obtained data show that along crack sides due to shear deformations grain edges become amorphous or the transition to higher-temperature modifications takes place.

Unfortunately, optical methods fail at estimation of the ultimate values of diffraction coefficients and to determine the silicon modification but they permit to determine the mechanism of plastic deformation of quartz materials. Under the microscope one can distincly see in quartz grains the lines of sliding (Fig. 2). They present rows of rounded holes as if from small bubbles (accumulations of vacancies). These defects are of dozens and hundreds of Angstrom in diam. A chain of such defects serves a nucleation for a crack. At considerable side pressures the lines of sliding are merged producing strips.

As a result of shear in the sliding plane a considerable amount of heat is produced. The temperature is increased above the point of phase transition. In [5–7] the process of plastic deformation is presented as a series of structural transformations of deformed material. In connection with the fact that heat is localized in the narrow zone of the order of $10^{-4} - 10^{-6}$ m [8] the phase transitions take place not over the bulk of grains, but in the individual points. In quartz at heating a series a chain of polymorphic transformations takes place [9]

$$\alpha\text{-quartz} \underset{\Theta = 2.4}{\overset{573°C}{\rightleftarrows}} \beta\text{-quartz} \underset{\Theta = 12.7}{\overset{870°C}{\rightleftarrows}} \beta\text{-tridimit} \overset{1470°C}{\rightleftarrows} \beta\text{-crystobalite} \overset{1723°C}{\rightleftarrows} \text{melt}.$$

The applied hydrostatic pressure results in the rise of temperature of the phase transition, and shear stresses, on the contrary, enhance the phase transitions [10–11], as a result, they are realized at lower temperatures. Incorporated defects and fluids also reduce transition temperatures.

It is to be noted that recently due to application of hydrostatic pressures in combination with shear stresses a number of new materials was developed as well as new types of transformations were observed. Extremely complicated transformations take place at very rapid rates (fractions and tenth of a second). Moreover, it has been found that these processes are in general, independent of temperature. It appears that in solids at high pressures and shear stresses molecules and atoms become highly movable and are apt to be activated. These processes are extremely complicated and need further investigations.

The transition $\alpha - \beta$-quartz is a transition of shear. No coordination bonds are broken, and the transition is reversible. At the $\alpha - \beta$-transition one observes abrupt variations of optical, structural, mechanical and thermal properties. However, at the reverse transition all properties are restored. Therefore, the fact of $\alpha - \beta - \alpha$-quartz transition can be stated only due to indirect indications, namely, by the form of cracks [12]. At temperatures greatly below the temperature point of $\alpha - \beta$-transition in quartz samples zigzag cracks appear with line segments parallel to faces of rhombhedron (Fig. 3). At $\alpha - \beta - \alpha$-rhombohedron transition curved cracks appear, as in amorphous solids, sealing and separation are not observed.

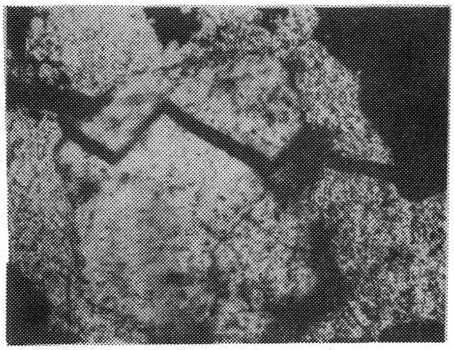


Fig. 3. Zigzag crack, quartz grain ×40, Nikoli+

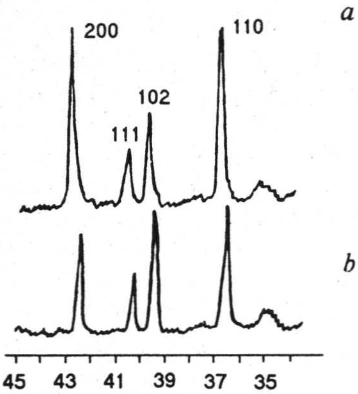
Considering that most cracks in sandstone samples at destruction in the volumetric stressed state are curved one can conclude that cracking takes place at the moment of $\alpha - \beta - \alpha$-transition.

Higher-temperature transitions are classified as the type of order-disorder transition. They can be stated as in samples of tridimite, crystobalite, amorphous silica and their transient modifications are observed.

In [13] after tests at 700°C and $15 \cdot 10^3$ MPa by scanning microscopy newly formed silica crystals were detected at edges of cracks. These edges were amorphous. However due to small sizes of crystals they were not identified. Therefore, we applied the diffraction technique to identify newly formed minerals. In the plane of shear destruction we took also some powder of dispersed material. To obtain powder we took some material from the undestructed part of the sample and crushed it in the mortar. Test powder diffractograms were taken with the aid of DRON-2, copper K_α-radiation. Comparison of diffractograms for Test Powder 1 and Test Powder 2 shows that in the plane of destruction considerable transformations take place. First, increase of interplane distances is detected and decrease of intensities of a series of quartz

Index of quartz reflection	Interplane distance		Line Intensity in the shear plane in persent to line intensity in the undestructed part of sample
	plane of destruction	nondestructed sample	
100	4.28	4.23	66
101	3.377	3.34	100
110	2.468	2.45	70
102	2.292	2.28	137
111	2.244	2.23	106
200	2.135	2.12	58
201	1.986	1.985	100
112	1.823	1.815	130
202	1.677	1.67	90
103	1.663	1.657	50
211	1.546	1.540	90
113	1.457	1.452	167
300	1.402	1.401	60
212	1.386	1.380	134
203	1.379	1.374	71
104	1.290	1.288	140
302	1.258	1.255	140
220	1.231	1.228	100
114	1.200	1.189	100
310	1.182	1.180	110
311	1.155	1.152	80
303	1.117	1.114	50
312	1.083	1.081	72
400	1.066	1.062	6

reflections (Table). The former cover all directions indicates at appearance (as a result of deformation) of dislocations with different Burgers vectors differing from dislocations that appear at crystallization of minerals, usually situated in one direction [14]. Reduction of intensity of lines 100, 110, 200, 103, 203 and disappearance of lines 300, 400, 303 can be explained by the fact of transition of the part of the sample to the amorphous state and disorder increase due to heat motions of atoms to the plane normal to the third order axis [15]. At the same time one observes growth of intensity of reflections 102,112, 113, 212, 104, 302 indicating at increase of order of the structure in these planes.



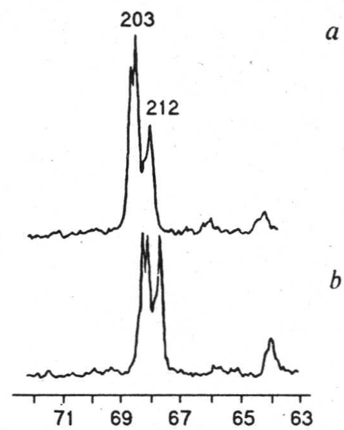


Fig. 4. Reflexes of quartz *a)* undestructed part of the sample; *b)* plane of destruction

Fig. 5. Quartz reflections, used for estimates of the crystalline degree, *a)* undestructed part of the sample; *b)* plane of destruction

As diagnostic reflections of quartz in [16] intensities of reflections of 110, 102, 111 are proposed. At diffractograms for quartz reflection 110 is always more distinct in comparison with reflection 102, and the height of peak 111 is greater half the height of peak 102. Exactly this is observed for Test Powder 2 (nondestructed part of the sample) (Fig. 4.). At diffractograms of the plane of shear the intensity of reflections 110 and 102 is practically the same, and peak 111 is half the height of peak 102, what is characteristic of chalcedics (latent-crystalline silica modification, of filament structure). This mineral being structurally disordered differs form quartz by tridimite-lines in diffractograms (4.52, 3.80 Å etc.), which can be explained by available tridemite-like regions [17]. Besides at diffractograms for the plane of destruction reflections of phases can be observed transient between quartz and amorphous silica: 4.75; 3.88; 3.40; 3.48; 3.12; 2.88; 2.43; 1.85; 1.35. These reflections are characteristicm for KT-opals, $SiO_2 - X_2$, $SiO_2 - Y$. All these minerals are either tridimites and crystobalites, which lost ordering, either aggregats of tiny grains of silica with platelet of filament structure. X-ray diagrams of them are similar to those for quartz and tridimite, but many lines are shifted towards increase of interplane distance.

For quantitative estimates of the crystalline degree of quartz and chalcedons Crystalline Index (CI) is applied, determined according to the intensity of line 212 [18]. Excess of 212 maximum over the background from large angles to the general height of the peak is considered. As is seen in Fig. 5. CI-index for quartz in the plane of shear is higher in comparison with the value for the nondestructed part of the sample (4.65 and 3.4 respectively). The smaller value is well agreed with the data [9], published for the sediment rock (1.3–3.2). The increased crystalline degree for chalcedons rings higher crystalline degrees are characteristic i.e., 3.3–4.7. The crystalline degree is an empirical characteristic and a criterion of defect content and the dispersion degree of crystals. Its increase (together with indications of disordering) one can explain by peligonization of crystals, i.e., crushing of crystals into smaller blocks with relatively regular structure. This process is due to redistribution of defects in the stress field.

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