VARIATIONS OF PHYSICAL PROPERTIES OF NEUTRON-IRRADIATED QUARTZ

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Radiation influence on the ensemble of physical properties (absorption, luminescence, and structural ones) of crystalline quartz has been investigated. The nonmonotone radiation kinetics of some optical and X-ray diffraction parameters in the regions of radiation-induced phase transition (PT) is established. A possible mechanism of crystal structure transformation under irradiation is discussed.

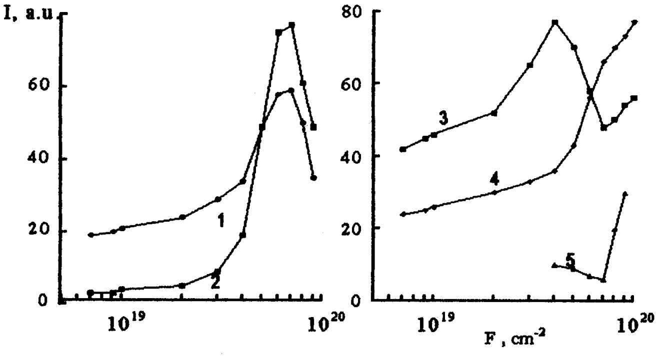
Due to a variety of properties, quartz, both in crystalline and vitreous forms, is widely used as one of the promising material in optical and electronic industry, in atomic and space techniques, in optoelectronics and radiophysics. Researches concerning the stability of its properties under the influence of various external factors (temperature, pressure, and radiation) have been started in the middle of the 20th century [1—3]. Heating and radiation actions on structural, elastic, electrical, optical, and some other properties of quartz have been considered there. But there is a discrepancy in those works concerning the mechanism of the structure reconstruction and the doses defining that step. Moreover, the features of the radiation-induced process of defect formation (RIPDF), relevant damages, and variations of a number of material properties at the PT point have not been studied enough, as well as a possible model of this phenomenon.

Few works dealt with investigations of stimulated reconstructions of the quartz structure. Moreover, the majority of them considers this cooperative process as a result of thermal treatment [1, 4—6]. In [1], an increasing of the point defect concentration near the PT temperature has been shown. In [4,5], making use of the isofrequency Raman scattering method, the temperature dependences of the order parameter relaxation time and the quartz mode frequency in the vicinity of PT have been determined. An evaluation of the rate of that process has been done in [6].

Recently, new works appeared concerning this problem [7—9]. But there is a small number of experimental works devoted to a systematic study of features of radiation-induced modifications of the ensemble of physical properties and the structure of "pure" single crystals of artificial quartz, possessing the same prehistory and geometry and irradiated under the same conditions by a variety of fast neutron fluxes.

This work aims at a complex investigation of the features of radiation-induced structure modifications of crystalline dielectric (α-quartz) and relevant changes of some of its parameters, as well as regularities in property modifications around the PT point. A number of spectroscopical [photoluminescent (PL), Raman, electron paramagnetic resonance (EPR), absorption], mechanical, and X-ray diffraction methods will be used. Lots of identical thin (thickness $l = 1$ mm) single-crystal plates were used as experimental objects. These plates were irradiated by beams of fast neutrons with various fluences, Φ, under the monitored conditions in the channels of a nuclear reactor WWR-SM.

The radiation kinetics of luminescence development in UV and visual spectrum ranges in α-quartz of high purity (the concentration of spontaneous impurities was less than $10^{-4}\%$) was studied. The dose dependences of PL band intensities, I, at 290 and 520 nm have a complicated behavior. In particular, sharp cusps appear at a threshold value ($\Phi_{\text{th}} = 4 \times 10^{19}$ cm^{-2}) in both dependences $I_{290}(\Phi)$ and $I_{520}(\Phi)$, while near $\Phi_c = 6.7 \times 10^{19}$ and 1×10^{-20} cm^{-2}, respectively, those dependences have maxima. According to results of [1—3,10,11], it was suggested that the cusps in the dose dependences of PL band light intensities are related to the acceleration of the RIPDF, that the defects of the type Si_2^0 (double-coordinated silicium) and O_2^- (interstitial oxygen) enhance their role in PTs of $\alpha - \beta$ and crystal—amorphous state (C—A) types, occurring at the doses indicated above, respectively. This is demonstrated by a dose curve *2* (see Figure), which corresponds to the generation of another intrinsic defect by fast neutrons, E'-center, and which was plotted making use of the results of the axial signal measurements in the EPR spectrum with $g_{\text{eff}} = 2.0013$ [12] for the same collection of specimens. A conclusion was made that the number of those defects (oxygen vacances) increases with the time of exposing the plates



Radiation kinetics features: $I_{\bar{3}303}(\Phi)$ — *1*, $I_{E'}(\Phi)$ — *2*, $I_{260}(\Phi)$ — *3*, $|\Delta\rho/\rho|(\Phi)$ — *4*, $B(\Phi)$ — *5*

in reactor channels, which corresponds to the generation kinetics of defects formed by gamma and neutron irradiations and by specimen heating [1—3]. This fact and the data of [7,8] concerning the accumulation of the mentioned EPR signal near T_c, evidence for their definite correlation.

As seen, there is a certain concentration of those defects which corresponds to a termination of the $\alpha - \beta$ transition in quartz. This conclusion is supported by coincidence of the maxima in $I_{E'}(\Phi)$ and $I_{\bar{3}301}(\Phi)$ dependences (curves *1* and *2*, respectively), the latter corresponding to a $\bar{3}301$ Laue reflection. On the basis of the revealed radiation kinetics and the data of [13], an idea of the micromechanism of (aggregation-involved) radiation-induced damage was put forward consisting in the following: if the threshold flux is exceeded, the radiation-stimulated aggregation of primary structure defects sharply accelerates up to a certain critical level, resulting in a PT.

Therefore, it was established that both the dose dependences for the $\bar{3}301$-reflection and EPR signal intensities have extrema in the range of fluences where a completion of crystal reconstruction by the high-temperature type is detected by X-ray diffraction. Indeed, it was found that $I_{3\bar{3}01}(\Phi_c)/I_{\bar{3}301}(\Phi_c) = 1$, and the unit cell parameter c calculated according to results of precise measurements of reflections 0006 ($2\theta = 117 \div 118°$) and 0003 ($2\theta = 50 \div 51°$) equals 5.460 Å at Φ_c. It happened that the values obtained in this work for the unit cell parameter of an irradiated crystal and for the ratio of the indicated reflection intensities correspond to those typical of the β phase obtained when heating a non-irradiated crystal to the PT temperature ($T_c = 573$ °C). This is confirmed by X-ray diffractograms recorded at DRON-3M which evidence for a drastical transformation of the 100-reflection profile ($2\theta = 20.90°$) in α-quartz irradiated by the indicated flux. This result correlates with the measurements on a heat-treated non-irradiated crystal carried out in [14], where such a modification of the 100 reflection is related to the formation of a stable phase close by parameters to β-quartz.

Thus, the results obtained confirm the assumption made earlier about both a feasible role of structural defects in the crystal structure reconstruction and the complexity of the very kinetics of formation of the state similar to the high-temperature modification of the substance. Using the X-ray diffraction method, we established a non-monotone character of the radiation-induced changes of the ratios of other reflection intensities in the PT region, e.g., reflections $3\bar{3}01$ and $4\bar{2}\bar{2}1$. The analysis of the dependence concerned showed that, in the first approximation, it obeys an exponential law

$$\frac{I_{3\bar{3}01}(\Phi)}{I_{4\bar{2}\bar{2}1}(\Phi)} = B^* \exp(b\Phi), \tag{1}$$

where $B^* = 2.6$ and $b = 0.15 \times 10^{-19}$ are fitting parameters. According to (1), the value for the $\alpha - \beta$ transition rate, K_1, was evaluated. It turned out that $K_1 = 2.3 \times 10^{-7}$ s^{-1}, which is close to a qualitative estimation obtained in [6]. A hypothesis concerning the availability of an extremum in the dependence of an intermediate phase fraction on neutron flux in crystals with polymorphism is really valid for our substance under investigation (α-quartz).

It should be noted that the discovered regularity (1) agrees well with one of the kinetic dependences obtained starting from the Prout—Thompkins equations [15] and evidences for that the studied nucleation process follows a sigmoid law with a short incubation period. Hence, in a general case, the kinetics of accumulation of a new radiation-induced phase of quartz is rather complicated.

A regularity in the generation of red PL (at 658 nm) in α-quartz was also traced. A substantial deviation of the dependence $I_{658}(\Phi)$ from the above-discussed $I_{290}(\Phi)$ and $I_{520}(\Phi)$ was found, because an anomaly in the form of "plateau" appeared at the stage of the intense $\alpha - \beta$ transition. For refining the possible origins of the revealed anomaly in the $I(\Phi)$ dependence, absorption optical spectra of irradiated crystals were studied in detail in the vicinity of 260—270 and 620 nm regions (curve *3*), which correspond to both excitation bands λ_{exc} of the lighting. A comparison of the optical densities of those bands $D_{260}(\Phi)$ and $D_{620}(\Phi)$ with $I_{658}(\Phi)$ dose dependences demonstrates the existence of an anomaly in the dose dependences of absorption bands in the analogous flux interval $(4 \div 7) \times 10^{19}$ cm^{-2}. Such a correlation of experimental data on the accumulation kinetics of point defects, which are of the non-bridge oxygen atom (NBOA, O_1^0) type [16] and are responsible for red luminescence, testifies to the influence of radiation and the PT on the luminescent-absorption characteristics of crystalline quartz and supports the viewpoint about the role of radiation-induced defects in the process of crystal structure modification.

As for the regularities of the radiation-induced defect generation, we obtained that the NBOA concentration grows at first along with Φ up to a certain value and, having reached it, decreases, which is traced in the interval $\Phi = (4 \div 7) \times 10^{19}$ cm^{-2}. Simultaneously, the reduction rate of the substance density sharply grows (see curve *4* which was plotted according to the results of hydrostatic weighting of specimens). These facts support the viewpoint [17] that broken Si—O—Si bonds assist the directional reconstruction of the structure, accompanied by a diminution of the number of breaks. Indeed, the analysis of the recorded PL spectra showed that, starting from the dose 1×10^{19} cm^{-2}, the increase of Φ by a factor of 2-, 4-, 5-, or 7 results in the change of the red luminescence intensity by a factor of 2, 3.5, 3.4, or 3.3, respectively.

In parallel with the studies of features of the processes of radiation-induced damaging and defect-formation in the dielectric, structural modifications of the same crystals were investigated by the method of X-ray scattering [11]. It turned out that, at $\Phi > \Phi_{\text{th}}$, a diffuse halo 1 with $\sin\theta/\lambda = 0.123$ Å appeared on X-ray patterns. Its peak position, B, shifts while the duration of the specimen irradiation increases. As is seen from curve *5*, for doses $(4 \div 7) \times 10^{19}$ cm^{-2}, the peak shifts little towards greater values. The comparison of curves *3* and *5* reveals an analogy of radiation-induced changes in the optical and X-ray diffraction signals in this interval.

The discovered feature of the $B(\Phi)$ dependence at $\Phi > 4 \times 10^{19}$ cm^{-2}, where the intense reconstruction into a state similar to β-phase of quartz is observed, may be a result of the increase of an average interatomic distance in a Si—O tetrahedron, which characterizes a loosening of small amorphous zones formed at the early stage of irradiation ($\Phi > \Phi_{\text{th}}$). Indeed, it was established [18] that, at $\Phi = 3 \times 10^{18}$ cm^{-2}, the density of amorphized microregions is less by 9% than that of initial α-quartz. At the same time, the experimental curve *4* demonstrates a drastic decrease of the quartz density at $\Phi > 4 \times 10^{19}$ cm^{-2}. In [19], a shortening of Si—O distances and a displacement of Si and O atoms in a direction peculiar to the β-phase were detected in quartz irradiated by the dose $(3 \div 4) \times 10^{19}$ cm^{-2}. Unfortunately, there is no information for $\Phi > 4 \times 10^{19}$ cm^{-2}, but we suppose that the noted behavior of changes of these parameters will remain further.

Thus, in the interval $\Phi = (4 \div 7) \times 10^{19}$ cm^{-2}, small dense disordered regions which are formed at small doses and gradually accumulated grow in size, interact with one another, and convert to less dense amorphous formations. All this is accompanied by a shortening of Si—O distances in the crystal environment, which apparently stimulates the PT. It is a preliminary conclusion about the possible mechanism of "pushing out".

In view of the results of [20] concerning the existence of a maximum in the magnetic susceptibility of quartz near $\Phi = 4 \times 10^{19}$ cm^{-2} and the data showing that, at this Φ, the dependence $D_{260}(\Phi)$ has a maximum and the dependence $I_{658}(\Phi)$ saturates, we can conclude that, at $\Phi > \Phi_{\text{th}}$, the formation rate of a relevant structure defect does decrease and the broken bridge bonds commence to repair, promoting the substance transformation into the more stable β-state. To explain the reason for the "plateau" appearance, two hypotheses have been put forward. As has been indicated above, making use of diffuse X-ray scattering at small fluxes in quartz, the emergence of amorphized microregions has been detected [18]. They comprise 4% of the specimen volume, and their density is by 9% less than that of the crystal, whereas at the full crystal amorphization, the density of specimens is by 14.5% less than that of α-quartz. This brings us to an idea that such microregions, emerging before Φ_{th}, have large concentrations of radiation-induced defects, including NBOAs. A possible explanation of this fact results from a thermodynamical consideration of the formation mechanisms of inherent defects in vitreous quartz at high temperatures [21]. In the experiment considered, for Φ in the interval $\Phi_{\text{th}} - \Phi_c$, a reconstruction of earlier formed dense and separated amorphous regions into less dense and overlapping ones seems to occur, which is indicated by changing the crystal density in this flux interval (curve *4*). According to [21], the visible "plateau" in the NBOA accumulation curve can be attributed to a compensation of the growth of the amorphous phase fraction by a decrease of the concentration of the defects, which are localized in the amorphized regions of specimens, so that their average concentration over the specimen remains constant.

The second hypothesis suggests an influence of either the PT process itself, since substantial modifications of a number of structural parameters, reflections, and crystal symmetry, or the Braun principle are observed.

According to the latter, an external action leading a system out of the equilibrium thermodynamical state, induces the processes aiming at reducing the results of the action. For example, when irradiating quartz, first, the intense processes of radiation-induced defect formation, structure damage, and emergence of amorphized regions are running up to Φ_{th}. Then, as Φ grows beyond the threshold value, the rates of those processes decrease, which results in a reduction of the growth rate of relevant defects.

Based on these facts and the revealed extremum for the number of E'-centers, the double-coordinated Si and interstitial oxygens, the following concept of the macroeffect under consideration has been advanced: the motive force of this process (the radiation-induced $\alpha - \beta$ transition) is a free energy δE transferred to the crystal by radiation-induced defects. Thus, on the basis of the data obtained, in the framework of field theory [23], we propose a percolation model of the radiation-induced $\alpha - \beta$ transition in quartz. Its main features are as follows. Let us suppose that amorphous domains are formed near the points of maximal temperature or maximal displacement. New phases, similar to β-phase, are formed and grow around those domains or around the clusters of primary defects, which is accompanied by a development of inner stresses. When old (α) and new (β) phases are in contact, an interface appears (an analog of domain wall). Those mobile interfaces are transition regions in a heterogeneous system. According to [23], the energy barrier for interface movement is reduced due to the interface smearing and the elastic interaction of phases. Furthermore, an increase of the interface mobility and the development of the PT turn out to be connected with the creation and migration of point defects. Therefore, an elastic deformation of phases reduces the thermodynamical stability of quartz and such formations with the increase of radiation dose, shifts the phase equilibrium point, and enhances the wall deformability. At last, when the motive force reaches a certain value at a certain critical dose Φ_c, the following condition is true:

$$E_\alpha + \delta E \geq E_\beta, \tag{2}$$

where E_α and E_β are the crystal lattice energies of the α and β phases, respectively, and δE is the energy transferred to the crystal lattice by defects. Then, a disruption of this wall occurs, a “leakage” occurs through the interface, and the whole crystalline residual transforms into a state similar to the β-phase, which completes the $\alpha - \beta$ transition. This is supported by an estimation showing that δE approaches, in this case, the thermal effect value, known at T_c.

It follows from the measured PL spectra that, after the completion of the $\alpha - \beta$ transition, the rate of NBOA-defect generation stepwise increases, which is traced up to $\Phi = 10^{20}$ cm^{-2}. At a further irradiation (up to 10^{21} cm^{-2}) of amorphized quartz, the $I_{658}(\Phi)$ dependence saturates.

Raman spectroscopy investigations discovered a new 608 cm^{-1} band in the crystal scattering spectrum, which appears at $\Phi = 5 \times 10^{19}$ cm^{-2}. Its intensity rises with Φ up to the completion of forming the amorphous phase. Let us suppose that the order parameter η is varied in the vicinity of a given defect. Then the size of the region disturbed by the defect will increase near the Φ_c dose, tending to infinity for $t = (\Phi - \Phi_c)/\Phi_c \to 0$. An increase of the size of the defect-deformed region governs the rising of the light scattering by defects, and, hence, the scattering intensity growth stipulated by fluctuations of the number of defects, which results in the appearance of the band 608 cm^{-1}, whose intensity grows with Φ, in the Raman spectrum of an irradiated quartz.

A question about the structure of centers responsible for the appearance of the 605—608 cm^{-1} band in the Raman spectra of vitreous quartz, finds no answer in the literature. This band is referred to local oscillations of atoms in intrinsic crystal defects formed at the breaking of Si—O bonds in plane circular structures, peroxide bridges, etc. The joint consideration of Raman spectra and $I(\Phi)$ dependences for PL centers, induced in quartz, evidences for that the 608 cm^{-1} band is related to a defect of the peroxide bridge (PB) type [16]. Such bridges emerge under aggregation of two point-like defects of the NBOA type in vitreous regions (we cannot exclude an extra channel for the accumulation of defects by an ordinary scheme of breaking the main Si—O—Si bonds). Therefore, the rise of the numbers of broken bonds, displaced atoms,and their aggregates leads, at a certain stage of irradiation, to a drastic enhancement of the light scattering by growing inhomogeneities of the metastable system, which manifests itself in the dynamics of Raman spectra and stimulates a quartz structure reconstruction, occurring seemingly by the seed mechanism.

An approximation of the dose dependence of the intensity $I_{608}(\Phi)$ of the relevant band (608 cm^{-1}) in the interval $\Phi = 4 \times 10^{19} - 1 \times 10^{21}$ cm^{-2} showed that the radiation kinetics of PB accumulation is well described by a power dependence

$$|R| = \text{const}\,|t|^{\Delta}, \tag{3}$$

where $R = I/I_b - 1$, $t = \Phi/\Phi_{\rm th} - 1$, and the power exponent $\Delta = 0.80 \pm 0.02$ is close to the index of correlation distance in the Shklovskii—de Gennes model for the 3-dimensional sphere packing problem.

It is remarkable that an approximation of the obtained dose dependence $I_{\rm 1h}(\Phi)$ of the halo 1 intensity showed the validity of (3) in this case as well, with $\Delta = 0.83 \pm 0.09$. The comparison of the empirical values obtained for the optical and X-ray diffraction parameters for the neutron-irradiated quartz reveals their coincidence.

The dielectric permittivity ε of quartz is another electric parameter sensitive to its structure. A non-monotone behavior was established for the function

$$f(\Phi) = 1 - \frac{\varepsilon_2(\Phi)}{\varepsilon_1(\Phi)}, \tag{4}$$

where $\varepsilon_2(\Phi)$ and $\varepsilon_1(\Phi)$ are the dose-dependent permittivities of crystalline and amorphous phases, respectively, both having cusp at the critical point $\Phi_{\rm th}$. An analogous radiation kinetics is also characteristic of the process of variation of the crystalline phase bulk fraction in the mixture. An approximation of the dielectric response as a function of the reduced dose of neutron radiation showed that it is described by a power dependence of the type (3) with $\Delta = 0.79 \pm 0.07$.

Let each sphere disposed in a crystal lattice node include an ensemble of disordered domains of the cluster type, which are, most probably, nuclei of the amorphous phase. Then, making use of the necessary condition for the presence of percolation in the sphere packing problem [22], we evaluated, in the first approximation, the critical concentration of those spheres $\Pi_{\rm cr}$ to be 0.7×10^{17} cm^{-3}. The evaluation showed that the condition [22]

$$L^{-3} < \Pi_{\rm cr}, \tag{5}$$

where L is a distance between clusters, is really fulfilled in our case ($L^{-3} = 3.0 \times 10^{15}$ cm^{-3}). This evidences for the validity, in the first approximation, of the idea of the appearance of percolation in quartz under exceeding the threshold dose, which is accompanied by the formation of an infinite cluster. In this case, we may assume that the intense growth of the amorphous phase takes place in the whole mixture of a solid solution. This growth is considered as a transformation of the solution into a disordered state at the contact areas of the crystal with the clusters of a new amorphous phase. We can conclude that, at this stage of the action of neutron irradiation, local amorphized regions complete the reconstruction of the crystalline quartz into the totally amorphous state.

Thus, the studied process of the C—A phase transition is very complicated in the general case and includes several channels where the reconstruction of the crystalline quartz structure is performed: namely, a continuous remelting of α-quartz at the points of temperature peaks or displacement peaks, a displacement mechanism according to a direct ($\alpha \to$ A) and indirect ($\alpha \to \beta \to$ A) schemes, and a threshold mechanism of radiation-induced crystal amorphization.

Thus, we have been experimentally investigated regularities inherent in the processes of radiation-induced defect formation and structure damaging in α-quartz irradiated by large neutron doses, as well as the variation of its properties and the formation of new modifications. A power-law dependence for radiation-induced changes of certain macroproperties of the material under study has been established. Possible channels for performing the phase transition have been considered, and a cluster model of the crystal amorphization has been suggested. The mechanism of the $\alpha - \beta$ transition in neutron-irradiated quartz has been discussed in the framework of the field model.

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ЗМІНА ФІЗИЧНИХ ВЛАСТИВОСТЕЙ НЕЙТРОННО-ОПРОМІНЕНОГО КВАРЦУ

І.Х. Абдукадирова

Резюме

Досліджено вплив радіації на комплекс фізичних властивостей кристалічного кварцу (абсорбційних, люмінесцентних, структурних). Виявлено немонотонну зміну радіаційних кінетик деяких оптичних і рентгенографічних параметрів. Обговорюються можливі механізми радіаційної трансформації кристалічної структури.