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FORMATION OF RADIATION DEFECTS AND STRUCTURAL TRANSFORMATIONS IN QUARTZ CRYSTALS IRRADIATED WITH CHARGED PARTICLES

Musoeva Gulrukh daughter of Sirojiddin 1
Mussaeva Malika Anvarovna 2
1 National University of Uzbekistan Named After Mirzo Ulugbek
Tashkent 100174, Uzbekistan,
E-mail: musoevaguli1618@gmail.com
2 Institute of Nuclear Physics, Academy of Sciences of Uzbekistan,
Tashkent 100214, Ulugbek Settlement, Uzbekistan
E-mail: mussaeva@inp.uz

ABSTRACT

The paper investigates the gamma-luminescence (GL) spectra of iron-doped Fe quartz crystals irradiated with protons and deuterons. Within the temperature range of 77–155 K, the GL spectrum is characterized by a complex band in the 450–520 nm region. Using the Gaussian deconvolution method, the key components were identified at 470 nm (recombination at E'1-centers) and 490 nm (annihilation of self-trapped excitons). It is established that an increase in beam current density and the mass of incident particles (transition from protons to deuterons) leads to an increase in the intensity of the 470 nm band, which is explained by the enhanced efficiency of displacement and ionization-induced creation of oxygen vacancies. The anomalous intensity behavior in the 77–85 K range is interpreted as the thermal activation of charge carriers from shallow traps, while the sharp decline in emission at 155 K is attributed to thermal quenching according to the Mott-Seitz model. It is shown that the Fe impurity modifies the energy structure of radiation-induced defects and affects their thermal stability.

KEYWORDS

Quartz, Fe-impurity, proton irradiation, deuterons, luminescence, E'1-centers, self-trapped excitons, thermal quenching.

Introduction

Quartz crystals are widely used in electronics, the optical industry, and in the production of special and technical glass [1]. A significant amount of natural quartz is used for smelting and as a feedstock in the synthesis of large, defect-free quartz single crystals with specified impurities and physical properties [2]. The study of the formation of point defects and structural transformations in crystals irradiated with charged particles (electrons, ions, protons) is one of the fundamental problems of solid-state physics and materials science [3–4, 5, 6]. Radiation exposure leads to the displacement of atoms

from the nodes of the crystal lattice and the formation of nonequilibrium defects, which significantly change the physical, mechanical, and optical properties of materials [6, 7]. In quartz crystals irradiated with a proton fluence of ≥ 4×10^{14} cm^{-2}, the formation of an amorphized phase containing E'_1 centers is observed [8–9]. According to modern concepts, the creation of point defects and structural transformations in quartz can occur due to both the ionization and bias components of the energy loss of high-energy ions [6, 10]. Since the contribution of the ionization and bias components depends on the energy, mass, and current density of the incident ions, the efficiency of the formation of radiation defects and new phase nuclei in crystals irradiated with different ions varies significantly [7,10].
An analysis of the literature data shows that the mechanisms of formation of point defects and structural transformations, as well as their dependence on the type and ratio of pre-radiation and radiation-induced structural defects in quartz crystals, have not been studied sufficiently [1,3]. In this regard, the aim of this work is a comparative study of the photo- and gamma-luminescence spectra of quartz crystals with *Fe* impurity, irradiated with protons and deuterons with fluences of 4×10^{14} and 10^{15} cm^{-2} at different beam current densities.

Research Objects and Methods

Quartz crystals were grown at the Russian Research Institute of Mineral Resources (RNIIMS, Alexandrov) in steel autoclaves under a pressure of 100 MPa in a 3% NaOH solution using the temperature gradient method at 340 ± 10 °C. Gamma-luminescence (GL) spectra were recorded at temperatures of 77 and 300 K using an SPM monochromator. Gamma-luminescence was recorded using an FEU-39 photomultiplier. GL was excited by 1700 R/s γ-rays from ^{60}Co (at the Institute of Nuclear Physics of the Academy of Sciences of the Republic of Uzbekistan). Samples were irradiated with various fluences of protons (Ep = 18 MeV), deuterons (Ed = 16 MeV), and electrons.

Results and Discussion

Analysis of the gamma-ray luminescence (GL) spectra of the studied quartz crystals in the temperature range of 77–300 K revealed a complex emission band in the visible spectrum. As shown in Fig. 1, the spectral profile is a superposition of two intense bands with maxima at 470 nm and 490 nm [1, 11–12]. According to the obtained data and deconvolution results, the nature of the observed emission is interpreted as follows:

1. The 490 nm band is due to the annihilation of self-trapped excitons (STEs), which is a fundamental property of the silicon dioxide structure under low-temperature excitation [3, 13–14].
2. The 470 nm band has a recombination nature and is associated with the capture of free charge carriers by luminescence centers associated with E'_1 centers (oxygen vacancies) [1,8].

It has been experimentally established that with increasing fluence and current density of the proton beam, competition between these channels is observed: the luminescence yield of the STE (490 nm, curve 1) decreases, while the intensity of the 470 nm band (curve 2) increases monotonically [1, 3]. Such dynamics indicate a high efficiency of non-impact creation of E'_1 centers with an increase in the ionization density in the tracks of charged particles [8].

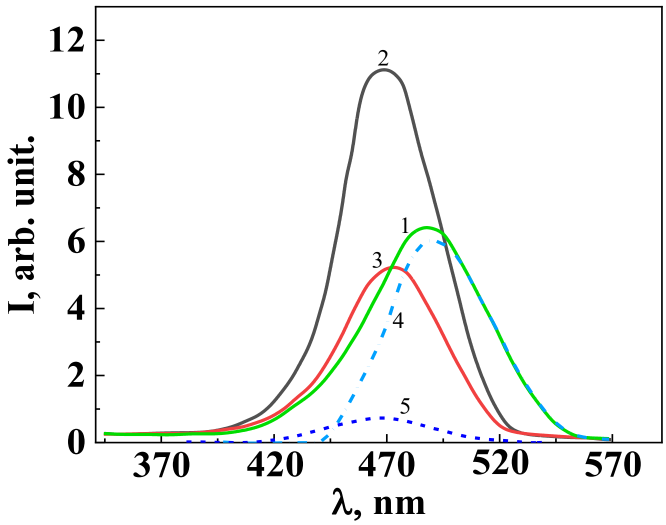

Fig. 1. Gamma-luminescence (GL) spectra of proton-irradiated quartz crystals with *Fe* impurity, recorded at 77 K (1), 85 K (2) and 155 K (3)

Of particular interest is a comparison of the efficiency of defect formation during irradiation with protons and deuterons. It has been established that, at identical fluences, the concentration of E'_1 centers in crystals irradiated with deuterons is significantly higher [1]. This is due to an increase in the cross section of both impact and non-impact creation of point defects with an increase in the mass of bombarding particles, which is consistent with the Kinchin-Pease theoretical model [7] and calculations of the energy losses of heavy ions in matter [6]. The temperature transformation of the spectra in the range of 77–155 K (Gaussian components: curve 4 and curve 5) demonstrates the competition between the processes of thermal activation of carriers from shallow capture levels and subsequent thermal quenching according to the Mott-Seitz model with an increase in temperature to 155 K [10]. The presence of iron impurity *Fe* acts as an additional factor in the stabilization of radiation defects, which affects the thermal stability of the isolated luminescence centers [13].

Figure 2 shows the change in the intensity of the 470 nm band in quartz crystals pre-irradiated with deuterons at a fluence of 10^{15} cm^{-2}, followed by additional γ-irradiation with varying doses. It was found that the intensity of the 470 nm band consistently increases with increasing γ-irradiation dose.

The observed increase in intensity is associated with the formation of additional radiation defects under the influence of γ-radiation. Additional γ-irradiation leads to an increase in the concentration of defect centers participating in luminescence processes, which is accompanied by an increase in the luminescence in the region of 470 nm.

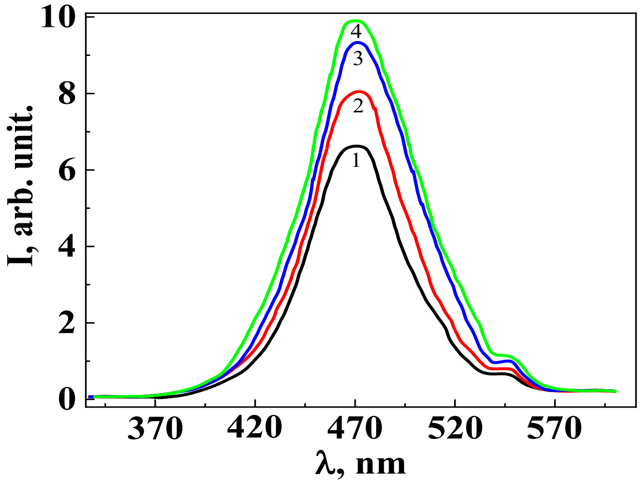

Fig. 2. Change in the intensity of the band at 470 nm in crystals pre-irradiated with a deuteron fluence of 10^{15} cm^{-2} (1), after additional γ-irradiation with doses of 10^6 R (2), 5×10^6 R (3) and 10^7 R (4)

Probably, γ-radiation activates both pre-existing defects formed under deuteron irradiation and creates new luminescence centers. According to [15], the formation of radiation defects in quartz under γ-irradiation can occur due to the non-radiative decay of excitons near already existing radiation-induced defects. An increase in the defectiveness of the structure contributes to an increase in the efficiency of this defect formation mechanism. The obtained results are also consistent with the data of [16], which showed that under radiation exposure, defect centers of the E'_1 type associated with amorphized regions of the structure are formed in quartz. Additional γ-irradiation likely promotes an increase in the concentration of such centers, which is reflected by an increase in the intensity of the 470 nm band. Thus, the increase in the 470 nm band intensity with increasing γ-irradiation dose indicates the accumulation of radiation-induced defects and the development of structural disorder in quartz.

Figure 3 shows the photoluminescence spectra of crystals irradiated with a proton fluence of 10^{15} cm^{-2} at beam current densities of 70 and 100 nA·cm^{-2}, after irradiation with an electron fluence of 3.7×10^{17} cm^{-2}, as well as the PL spectra of crystals irradiated with a deuteron fluence of 4×10^{14} cm^{-2}.

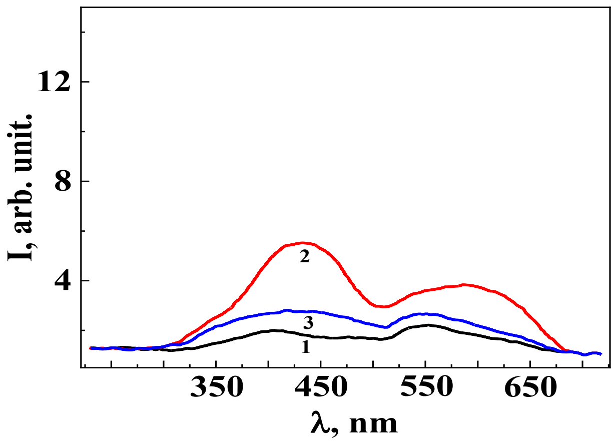

Fig. 3. PL spectra of crystals pre-irradiated with a proton fluence of 10^{15} cm^{-2} at beam current densities of 70 (1) 100 nA·cm^{-2} (2) after exposure to an electron fluence of 3.7×10^{17} cm^{-2}, and irradiated only with a deuteron fluence of 4×10^{14} cm^{-2} at 70 nA·cm^{-2} (3)

Figure 3 shows the photoluminescence spectra of quartz crystals after various types of radiation exposure (curves 1–3). Curve 1 corresponds to a sample irradiated with protons with a fluence of 10^{15} cm^{-2} at a beam current density of 70 $nA{\cdot}cm^{-2}$. The spectrum contains relatively weak photoluminescence bands in the regions of 420–470 nm, 520–580 nm, and 600–650 nm, which indicates the initial stage of the formation of radiation defects, primarily oxygen vacancies. According to [15], at a relatively low degree of defectiveness of the quartz structure, the formation of radiation centers is associated mainly with local disturbances of the oxygen sublattice. Curve 2, corresponding to a sample pre-irradiated with protons followed by electron irradiation with a fluence of 3.7×10^{17} cm^{-2}, is characterized by the highest intensity in the entire spectral range studied.
In the short-wavelength region ($\lambda \approx$ 420–470 nm), an intense band associated with oxygen-deficient centers (ODC) is observed. A significant enhancement of this band indicates a significant increase in the concentration of oxygen vacancies. In the region of 520–580 nm, a broad band appears due to the presence of E'-centers and other radiation-induced defects. In the long-wavelength region (600–650 nm), a contribution from complex defect centers formed under high radiation exposure is observed. An increase in the intensity of all bands under combined irradiation indicates a synergistic effect of the sequential action of protons and electrons. This behavior is consistent with the results of [16], where it was shown that an increase in the fluence and current density of the irradiating particles leads to an increase in the concentration of E'_1-centers associated with amorphized regions of quartz. Additional electron irradiation promotes the activation of previously formed defects and the formation of new luminescence centers. Curve 3 corresponds to a sample irradiated with deuterons with a fluence of 4×10^{14} cm^{-2} at a current density of 70 $nA{\cdot}cm^{-2}$. The spectrum also contains the indicated bands, but their intensity is significantly lower compared to curve 2. This is due to the lower irradiation fluence and, accordingly, a lower concentration of induced defects, despite the higher deuteron mass. Thus, a comparative analysis of the photoluminescence spectra shows that oxygen-deficient centers, E' -centers, and complex radiation defects are formed in all the studied samples. The maximum concentration of defect centers is achieved with sequential proton and electron irradiation (curve 2), while curve 1 reflects the initial stage of radiation defect formation.

Conclusion

This study investigated radiation-induced defect formation in *Fe*-doped quartz crystals exposed to protons, deuterons, γ-radiation, and electrons. It was found that the gamma-luminescence and photoluminescence spectra depend significantly on irradiation conditions and measurement temperature. An increase in temperature was shown to change the intensity of the gamma-luminescence band at 470 nm, which is associated with the thermal activation of radiation-induced defect centers. Additional γ-irradiation of pre-irradiated crystals causes a monotonic increase in the intensity of the 470 nm band due to the formation of new defects and an increase in the concentration of luminescent centers.
It was found that the combined action of protons and electrons is the most effective factor in radiation-induced defect formation. This process leads to the formation of oxygen-deficient centers, E'-centers, and complex defect states associated with partial amorphization processes in the quartz structure. Increasing the beam current density promotes increased structural disorder and an increase in photoluminescence intensity.

These results are consistent with published data [15, 16] and confirm the significant role of radiation-induced defects in the formation of quartz's luminescent properties. These results can be used in the development of radiation-resistant quartz materials, as well as for further study of the mechanisms of radiation-induced defect formation and structural transformations in dielectric crystals.

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