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## X-Ray And Phase Analysis Of $\text{SiO}_2$ Glass

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### ABSTRACT

The formation of nanosized particles in pure silica glass irradiated in water in the reactor core with fast neutrons up to  $10^{19} \text{ cm}^{-2}$  and gamma radiation has been studied by optical spectroscopy and X-ray diffraction. Irradiation with neutrons leads to the destruction of the initial mesostructure of  $\alpha$  - and  $\beta$  - quartz with dimensions of 1.7 and 1.2 nm and the formation of cristobalite and tridymite nanocrystallites with dimensions of 16 and 8 nm in displacement thermal peaks.

### KEYWORDS

Pure quartz glass of optical grades KI and KV, gamma irradiation, neutron irradiation, X-diffraction spectra.

### INTRODUCTION

Currently, X-ray phase analysis (X-ray, or X-ray diffraction) is the most common of the diffraction analysis methods. It should be noted that diffraction methods are used to

study the structure of not only crystalline solids, but also liquids and glasses. Radiation defects in  $\text{SiO}_2$  materials have been intensively studied for half century, since they exist in

various crystalline and glassy modifications, which are widely used in technology and nuclear power [1]. It is known that fast neutrons create displacement cascades. Due to the mixed covalent - ionic nature of the Si-O chemical bond, point radiation defects can arise by the radiolysis mechanism under purely ionizing irradiation as result of nonradiative recombination of bound electron – hole pairs [2, 3]. Research [4] has shown that under the influence of  $\gamma$ -radiation  $^{60}\text{Co}$  with most of the quartz glass is colored, and it can be used in dosimetry. Absorbance intensity at wavelength  $\lambda=550$  nm increases almost linearly with an increase in the absorbed energy  $\gamma$ -radiation dose to  $10^5$  Gy, which indicates the possibility of using quartz glass for the brand CI dosimetry  $\gamma$ -radiation.

Earlier, we discovered the growth and dissolution of nanoparticles in  $\text{SiO}_2\text{-BaO}_x$  glass under  $^{60}\text{Co}$ -gamma irradiation by methods of X-ray diffraction and optical spectroscopy. Nanocrystals and their sizes have been determined [5].

We have experimentally established [6, 7] that when glass is irradiated in the thermal column of an atomic reactor or in  $^{60}\text{Co}$  gamma device, phase transformations of  $\text{SiO}_2$  nanocrystallites cristobalite into tridymite, BaO into  $\text{BaO}_2$ ,  $\text{BaSiO}_3$  occur. The localization of charge carriers at the interphase boundaries causes brown color, the weakening of the luminescence intensity, and a decrease in microhardness as the result of the removal of mechanical stresses.

**The aim** of this work was to determine the influence of structural changes which are

caused by irradiation with ionizing rays and gamma irradiation in a reactor in  $\text{SiO}_2$  glass.

## EXPERIMENTAL RESULTS

**Samples** - There were pure silicate (quartz) glass of optical grades KI (transparent in the IR region) and KB (transparent in the UV region), manufactured at LOMO (Russia).

**Gamma irradiation** - Samples were wrapped in Al foil for isolation from air, irradiated in a dry channel at 320 K with  $^{60}\text{Co}$  gamma quanta at the dose rate of 406 R/s (energies 1.17 and 1.32 MeV) up to  $10^9$  R and irradiation in a reactor of  $10^{19}$  n/cm<sup>2</sup>.

**X-ray and phase analysis:** To determine the structure of the near-surface layer of unirradiated and irradiated samples, X-ray diffraction spectra were recorded under identical conditions on an X-ray diffractometer (XRD Empyrean, PANalytical). X-ray fluorescence analysis was carried out on the multichannel analyzer with Ge - detector in order to determine the total elemental composition averaged over the near-surface layer by selecting radioactive sources of X-ray excitation of k - lines of elements to the depth of the half-absorption layer of radiation.

Since the energy of X-ray radiation of 0.154 nm from copper tube is less than 50 keV, then it penetrates into the substance to a depth of less than 100  $\mu\text{m}$ , depending on the density and atomic number. Therefore, X-ray analysis is carried out in thin near-surface layer, and not in the entire volume of the substance.

Technical characteristics of the X – ray diffractometer Empyrean (PANalytical) are shown in the table:

Configuration - Reflection - Transmission	Anode material Cu
Goniometer Theta/Theta radius [mm] 240.00, scan axis –Gonio	K-alpha1 [Å] 1.54060 K-alpha2 [Å] 1.54443 K-A2/K-A ratio 0.50000
Minimum scan step 2 Theta 0.0001	K-Beta [Å] 1.39225 cut by Ni filter
Start [2θ] - 5.0039, end - 139.9919, step [2θ] 0.0070, step time [s] 37.9950	X-radiation generator settings Current 40 mA, voltage 45 kV
Slit divergence [°] 0.4354	Sample thickness [mm] no more than 3
Distance from focus to slit [mm] 100.00	Sample length [mm] 10.00- 30.00
Incident Beam Monochromator - No	Measurement temperature [°C] 25.00±5
Measurement of X-spectra using the Data Collector software	Determination of the phase composition according to the PDF-2 database 2013
Stage with sample rotation in horizontal plane with the minimum step of 0.1	X-ray processing (background level, peak identification, peak profile analysis) using the High Score program

## RESULTS AND ITS DISCUSSION

The investigated grades of pure silicate (quartz) glass of optical grades KI and KV

before and after gamma irradiation are shown in Fig. 1.

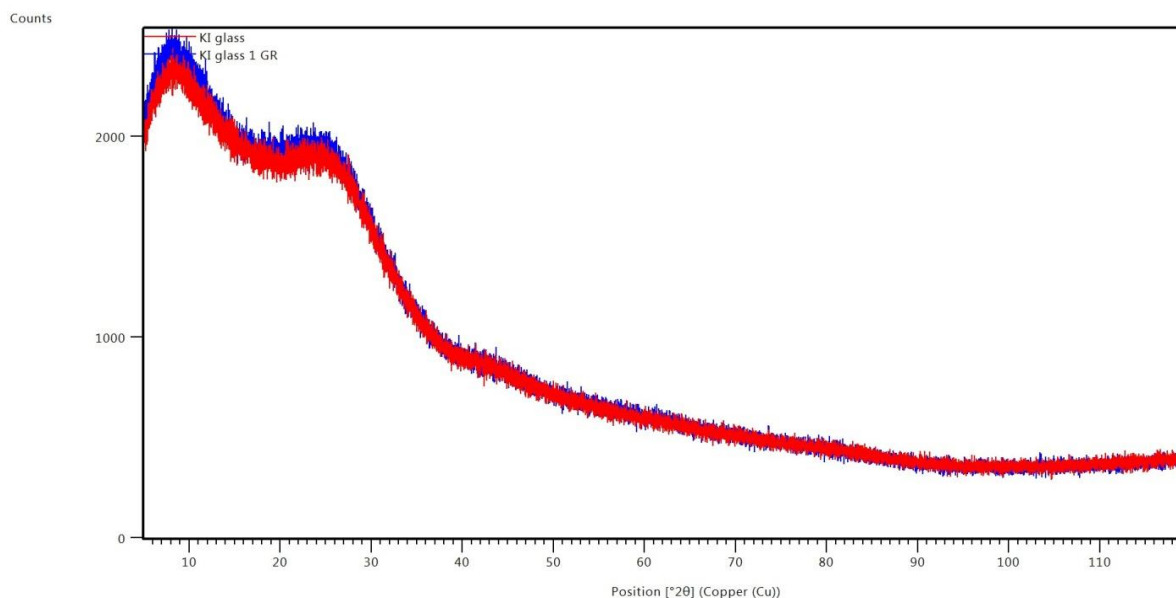
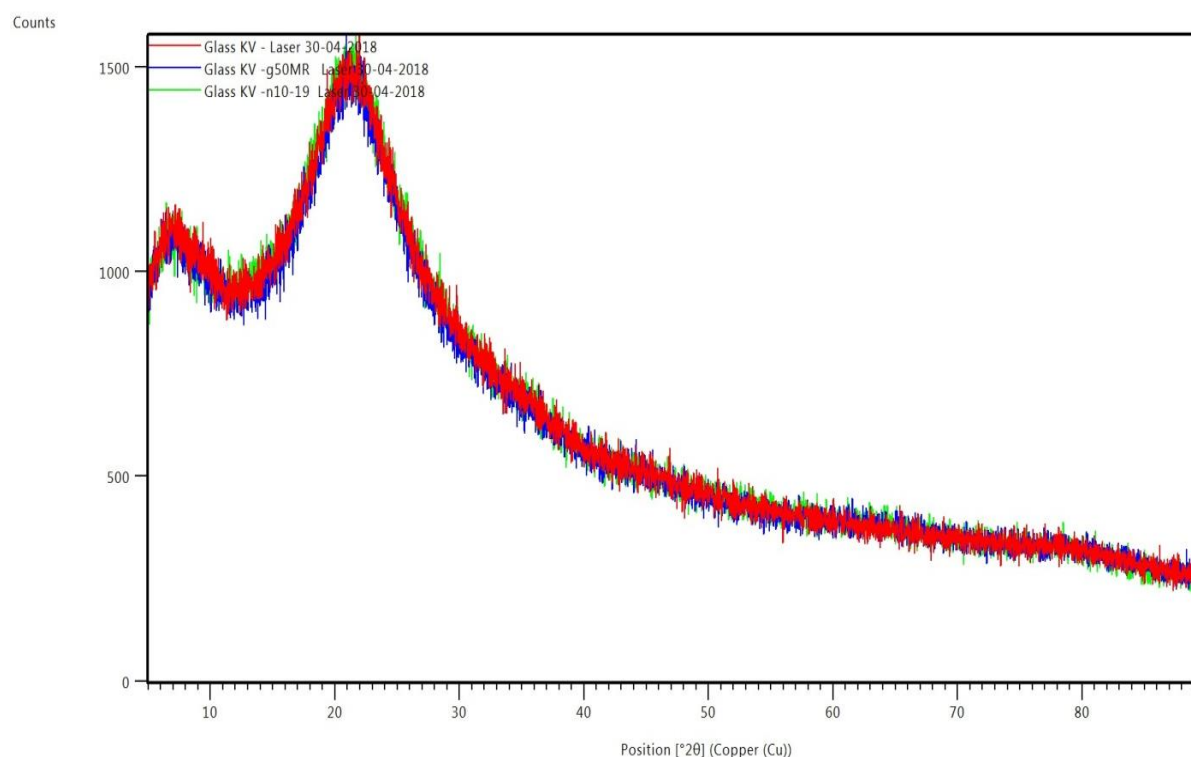


Figure 1. X-diffraction spectra of KI quartz glass: red - unirradiated, blue - gamma irradiated with a maximum dose of  $10^9$  R

Intense small angle scattering contains two bands,  $8^\circ$  and  $25^\circ$ , which are used to determine the sizes of cells with short range crystal order in the glass network - tetrahedron and octahedron. Reflections of crystalline phases of quartz, cristobalite and tridymite above diffuse bands after prolonged gamma irradiation can be distinguished, which indicates radiation-induced crystallization of nanoparticles due to the absorption of excess energy from radiation. In the case of gamma irradiation, where the heating of the sample does not exceed  $80^\circ\text{C}$ , the mechanism of cold

crystallization (atomic ordering) can be proposed, which absorbs the bulk of the radiation energy. Leaving little for the formation of defects. Then this can explain the high radiation resistance of this glass.

In fig. 2 shows the X-diffraction spectra of KV quartz glass, unirradiated and gamma irradiated with a dose of 50 MR and irradiated in a reactor of  $10^{19} \text{ n/cm}^2$ .



**Figure 2 - X-diffraction spectra of KV quartz glass: red - unirradiated; blue - gamma irradiated with a dose of 50 MR; salad - irradiated in a reactor  $10^{19} \text{ n/cm}^2$**

In contrast to KI glass, octahedral cells predominate in KV glass, although of smaller sizes, which are responsible for the  $22^\circ$  band, practically did not change the microstructure of the glass network and the scattering background on nanoscale cells of the glass

network. It can be seen that large doses of gamma and neutron irradiation give practically the same weak effect of scattering from radiation-induced point defects.

## CONCLUSION

Silicate glasses KI and KU consist of two types of cells, differing in size from 0.5 to 1.5 nm. After irradiation, weak reflections of nanocrystalline phases responsible for UV absorption appear in them. The fact that the absorbed radiation energy is spent on the crystallization of SiO<sub>2</sub> determines their high radiation-optical resistance.

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