Structural Changes in Silica Glass under the Action of Electron Beam Irradiation: The Effect of Irradiation Dose

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Received August 20, 2020; revised September 29, 2020; accepted December 4, 2020

Abstract—Structural changes in silica glass, produced by electron irradiation, with electrons energies of 50 keV and doses of 5–80 mC/cm² were studied by Raman spectroscopy in frequency ranges of 300–550 and of 700–1500 cm^{−1}. It is shown that for irradiation doses less than 5 mC/cm² the decrease of siloxane rings concentration in glass takes place. The further increase of dose results in the increase of siloxane rings concentration in glass network after irradiation. And for doses of more than 40 mC/cm² the whole destruction of glass structure and its structural units takes place. The obtained results for silica glass were compared with similar results for crystalline quartz.

Keywords: silica glass, glass structure, electron beam, Raman spectroscopy

DOI: 10.1134/S1087659621020140

INTRODUCTION

Focused electron beam with relatively low electrons energies (less than 100 keV) is a powerful tool for local modification of optical properties of optical materials, such as glasses and crystals. By electron irradiation the crystallization processes in glasses can be initiated [1, 2]. Metal nanoparticles (Ag, Au, Cu, Na, K, Li etc.) can be formed locally under the surface of metal-containing glasses and crystals [3–7]. Optical and plasmonic waveguides can be written by scanning electron beam on glass or crystal surfaces [3, 8–10]. Electron beam irradiation of glasses can enhance non-linear optical properties of glasses [11] and produce luminescent centers in them [6, 7]. The choice of electron energy makes possible to dissolve thin metal films in glasses or to form metal films on the surfaces of metal-containing glasses [12, 13]. The advantage of electron beam with relatively low electrons energy is that it can be focused into a spot with the size less than 10 nm. It makes possible to fabricate nanostructures in glasses and crystals for integrated optics, nanophotonics and nanoplasmonics.

The main processes, which take place during focused electron beam action on glasses are the following: (i) appearance of negatively charged area under the sample surface, formed by thermalized electrons [3, 14]; (ii) field migration of positive and mobile metal ions to the negatively charged area, thus producing spatial re-distribution of glass components and local change of its chemical properties [3, 15]; (iii)

breaking of chemical bonds by fast electrons, thus producing structural defects and changing the structure and other properties of glass. To study the features of the third process the most convenient material is pure silica glass without any dopants.

The influence of ionizing radiations (such as X-rays, γ -rays, heavy ions beams, synchrotron and UV irradiation, etc.) on structural and other properties of glasses was studied in [16–35]. In [35] structural changes in silica glass after electron irradiation with electrons energy 2.5 MeV was studied by methods of Raman spectroscopy. There are very few works discussing the influence on glasses of electrons with relatively low energies (1–100 keV), though this interval of electrons energies is most promising for the use in nanotechnologies. And it is important for further investigations to study the influence of electron irradiation on “pure” SiO₂, such as silica glass.

The goal of the present work was the study of the influence of electron irradiation dose, for electrons with energy of 50 keV, on the structural changes of silica glass by methods of Raman spectroscopy.

EXPERIMENTAL

In our experiments we used polished plates of optical silica glass KU-2 with the size of 15 × 15 mm² and 3 mm thick. The electron irradiation was carried out in a JEED-2 scanning electron microscope. The electrons energy E was 50 keV, the irradiation doses D were

varied in the range of 5–80 mC/cm² for electron current density of 50 μ A/cm². The electron irradiation was carried out at a room temperature, but the calculation have shown that near-surface glass layer during electron irradiation for $E = 50$ keV and $D = 50$ mC/cm² is heated up to 150°C. The calculation was performed by solving of non-stationary equation of thermal conductivity with the assumption that all the energy of primary electrons is transformed to heat in near-surface glass layer of 50 μ m thick. The electron beam diameter on the glass surface was 1.5–2 mm for the convenience of optical measurements. Aluminum films of 80 nm thick were deposited on the samples surfaces for removing the surface charge, which appears on a glass surface during electron irradiation. After irradiation, these films were removed by etching in an aqueous solution of KOH.

Raman spectra were measured using inVia Raman microscope spectrometer (Renishaw) at room temperature. It is known that some types of radiation defects are non-stable and disappear after some time. Preliminary studies have shown that strong changes in Raman spectra stop approximately 10 days later after electron irradiation. So, spectral measurements were performed after the mentioned time interval.

EXPERIMENTAL RESULTS

Figure 1a (curve 1) shows the Raman spectrum of silica glass before electron irradiation. One can see that spectrum consists of three main bands: the structured and intense band in frequency range of 200–700 cm⁻¹ (it can be decomposed by 4 Gaussians), weak bands with maxima at 800 and at 1075 cm⁻¹. The band at frequencies less than 200 cm⁻¹ is the superposition of Boson peak, typical one for vibrations in disordered media [36, 37], and Raleigh scattering. According to [38–40] the band with maximum at $\nu = 1075$ cm⁻¹ can be assigned to Si–NBO (NBO – non-bridging oxygen) stretching mode in Q³ unit (see Fig. 1b). The band at $\nu = 800$ cm⁻¹ is related to the symmetric stretching mode of anionic structural unit (SiO₄)⁴⁻. The bands with $\nu = 600$ cm⁻¹ can be related to the symmetric oxygen breathing vibration of four-fold and three-fold siloxane rings, consisting of SiO₄ tetrahedra (see Fig. 1b). The bands at about 420 and 465 cm⁻¹ can be attributed to vibrations of Si–O–Si bonds in four-fold, five-fold, and six-fold siloxane rings. The band at $\nu = 330$ cm⁻¹ can be attributed to Si₂O₄ in 3D-framework [41].

For the comparison in Fig. 1a are shown Raman spectra of sodium-containing silicate glass with the composition Na₂O–SiO₂–CaO (curve 2) and of crystalline α -quartz (curve 3). One can see that for silicate glass the ratio of Raman bands intensities at $\nu \approx 400$ cm⁻¹ and $\nu \approx 1000$ cm⁻¹ differs from the ratio of the same bands intensities for silica glass. It indicates the con-

siderable increase of concentration of glass network defects in silicate glass, such as Q² and Q³ (see Fig. 1b). Raman spectrum of crystalline α -quartz consists of two main bands: narrow band at $\nu = 465$ cm⁻¹ and wide structured band in frequency region of 700–1100 cm⁻¹. The first band can be attributed to Si–O–Si stretching-bending modes in 6-fold siloxane rings [42]. The second band can be attributed to the point defects in crystalline quartz, similar to Si–NBO.

After electron irradiation no visible changes in irradiated areas of the samples were observed. Figure 2 shows Raman spectra of silica glass in frequency range of 300–550 cm⁻¹ before and after electron irradiation with different irradiation doses. Figure 3 shows the influence of irradiation dose on Raman bands intensity for $\nu = 465$ and 420 cm⁻¹. It can be seen from Figures that for doses range of 0–5 mC/cm² the decrease of bands intensity in their maxima takes place. For $D = 10$ –40 mC/cm² the increase of bands intensity is observed, and they became more structured. After irradiation with $D = 10$ mC/cm² new band with maximum at $\nu = 375$ cm⁻¹ appears. For irradiation dose $D = 40$ mC/cm² the bands intensity returns to the initial state. When D exceeds 40 mC/cm² the intensity of bands decreases with the increase of irradiation dose. The change of bands intensity is accompanied by the spectral shift of their maxima. The dependencies of intensity on irradiation dose for $\nu = 465$ and 420 cm⁻¹ are similar in the limit of measurements error (Fig. 3).

Figure 4 shows Raman spectra of silica glass in frequency range of 700–1300 cm⁻¹ before and after electron irradiation with different irradiation doses. It can be seen from Fig. 4 that there are, at least, 6 Raman bands with different intensities in this spectral region. It indicates the presence in silica glass of several types of defects with different properties [38–40]. The dose of electron irradiation effects on all bands, changing their intensity and, in some cases, produces spectral shift of the bands. Below we discuss the influence of electron irradiation dose only on three bands, which can be accurately attributed. These bands have maxima at $\nu = 800$, 1000 and 1075 cm⁻¹. The first band ($\nu = 800$ cm⁻¹) corresponds to the symmetric stretching mode of anionic structural unit (SiO₄)⁴⁻ [39]. The second band with maximum at $\nu = 1000$ cm⁻¹ is the superposition of two bands: Si–NBO stretching mode in Q³ unit and Si–BO vibration mode in not fully polymerized structural units Q³ or Q². The third band with maximum at $\nu = 1075$ cm⁻¹ can be assigned to Si–NBO stretching mode in Q³ unit.

Figure 5 shows the influence of irradiation dose on these Raman bands intensities in maximum. One can see that the increase of irradiation dose from 0 up to 10 mC/cm² results in the considerable increase of

Fig. 1. Raman spectra of silica glass (1), sodium-containing silicate glass (2) and crystalline α -quartz (3) before electron irradiation. Green dashed curves are the decomposition by Gaussians. The spectra of silicate glass and α -quartz are shifted up (a); some structural units of silica and silicate glasses. BO—bridging oxygen, NBO—non-bridging oxygen (b). Adapted from [38–41].

bands intensities. The further increase of irradiation dose is followed by the decrease of intensity for all three bands. The amplitudes of the bands for $D = 80 \text{ mC/cm}^2$ are less than in non-irradiated sample.

For the comparison the sample of crystalline α -quartz was also irradiated by electrons in the similar conditions. Figure 6 shows the influence of irradiation dose on Raman band intensity at $\nu = 465 \text{ cm}^{-1}$ (see also Fig. 1a, curve 3). It can be seen from the Figure that electron irradiation of crystalline quartz results in monotonous decrease of the band intensity with the increase of irradiation dose. The most considerable changes in band amplitude are observed for $D < 20 \text{ mC/cm}^2$. The influence of electron irradiation on Raman band in frequency range of $700\text{--}1100 \text{ cm}^{-1}$ (see Fig. 1a, curve 3), which is connected with crystalline defects, is very weak and does not exceed measurements error.

DISCUSSION

It must be noted that structural changes in glass take place not only during electron irradiation, but also after irradiation. The reason of it is that some radiation defects, which appear in during electron irradiation, are non-stable [43], and after electron irradiation the processes, involving them, can continue in glass. Let's consider the main processes, which cause structural changes during electron irradiation specifically in silica glass. The calculations (by Monte-Carlo method) have shown that maximum of energy losses for electrons with $E = 50 \text{ keV}$ is located in depth of $25\text{--}30 \text{ }\mu\text{m}$ under the glass surface. So, all the changes in glass during electron irradiation are located in a subsurface layer of that thickness. As it was mentioned in Introduction the main processes, which take place in glasses during electron irradiation, are the break of chemical bonds of glass network and the field migration of positive mobile ions to the nega-

Fig. 2. Raman spectra of silica glass in the frequency range 700–1300 cm^{-1} . Irradiation doses D in mC/cm^2 : 1—0, 2—5, 3—10, 4—15 (a); Irradiation doses D in mC/cm^2 : 5—30, 6—40, 7—60, 8—80 (b). Dashed lines are spectral position of bands, analyzed below.

tively charged region, formed by thermalized electrons. The components of silica glass, Si and O in ionic state, have very low mobility at room temperature, so there is very low probability of their spatial redistribution during electron irradiation. Thus, the main process, which causes the changes in silica glass structure, is the break of chemical bonds of glass network by fast electrons. It is known that the binding energy of Si—O bond is approximately 8 eV. So, electrons with this or higher energies can break bonds in siloxane rings, decreasing their concentration in glass. This is confirmed by the decrease for $D \leq 5 \text{ mC}/\text{cm}^2$ of intensities of Raman bands at $\nu = 420$ and 465 cm^{-1} , which can be attributed to vibrations of Si—O—Si bonds in siloxane rings (see Fig. 3). At the same time the concentration of glass network defects, such as Q^2 , Q^3 and $(\text{SiO}_4)^{4-}$, for $D \leq 10 \text{ mC}/\text{cm}^2$ increases (see Fig. 5).

As for low irradiation doses the concentration of radiation induced defects is low, and they are located far from each other, the restoration of siloxane rings after electron irradiation is difficult. But it can be seen from Figs. 3 and 5 that for higher doses the increase of siloxane rings concentration and the decrease of defects concentration take place. It is evident that the higher is the irradiation dose the higher concentration of defects will appear in glass. And at high concentration of defects it is easier for them after electron irradiation to form new siloxane rings, thus restoring the structure of glass network. When electron irradiation dose exceeds $40 \text{ mC}/\text{cm}^2$ the decrease of Raman bands intensity at 420 and 465 cm^{-1} is observed. This can be explained by the destruction of Q^2 , Q^3 and Q^4 structural units, which makes the restoration of siloxane rings impossible.

Fig. 3. Influence of irradiation dose on Raman bands intensity at $\nu = 465 \text{ cm}^{-1}$ (1) and 420 cm^{-1} (2).

Strong structural changes in silica glass after electron irradiation for large irradiation doses are confirmed by considerable change of glass chemical etching rate in the irradiated zone. Figure 7 shows the photo of silica glass surface near irradiated zone, made by optical microscope. In this case electron irradiation dose was $D = 80 \text{ mC/cm}^2$, chemical etching was performed in HF (10%) during 3 h. One can see that after etching the pit appears in the irradiated zone. The rate of etching in this region increased approximately by 2 times.

It can be seen from Figs. 2 and 4 that electron irradiation results in spectral shift of some Raman bands, as in spectral range of siloxane rings vibrations, so in the spectral range of bonds vibrations in glass network defects. In the first case, it can be explained by the change of number of elements in siloxane rings, restored after electron irradiation, in comparison with

Fig. 4. Raman spectra of silica glass in the frequency range $700\text{--}1300 \text{ cm}^{-1}$. Irradiation doses D in mC/cm^2 : 1—0, 2—5, 3—10, 4—15. Inset—curve 1 (a); Irradiation doses D in mC/cm^2 : 5—30, 6—40, 7—60, 8—80 (b). Dashed lines are spectral position of bands, analyzed below.

Fig. 5. Influence of irradiation dose on Raman bands intensity at $\nu = 800$ (1), 1000 (2), and 1075 cm^{-1} (3).

pristine glass. The change of siloxane ring size leads to the change of Si–O chemical bond length. The other reason is the appearance of Si–O–O–Si units in restored siloxane rings, as a result of connection of two NBO's. In the second case, at high concentration of radiation-induced defects the interaction between them can take place. This can influence on chemical bonds lengths in defect structural units, such as Q^2 and Q^3 .

The dependence of Raman band intensity on electron irradiation dose at $\nu = 465 \text{ cm}^{-1}$ for crystalline α -

Fig. 6. Influence of irradiation dose on Raman band intensity at $\nu = 465 \text{ cm}^{-1}$ for crystalline α -quartz.

quartz differs from the similar dependence for silica glass: the Raman band intensity monotonously decreases with the increase of irradiation dose (see Fig. 6). It can be explained by the fact that crystalline quartz is more static system than silica glass, and dynamic processes after electron irradiation, including siloxane rings restoration, occur at room temperature too slow. The weak influence of electron irradiation on Raman band, connected with defects in quartz, is caused by the difference of irradiation induced defects types, in comparison with silica glass.

Fig. 7. Photo of silica glass surface near the irradiated area after chemical etching. Scale is 1 mm.

The obtained results can be compared with the results, described in [35], where silica glass was irradiated by electrons with energy of 2.5 MeV and after that was studied by Raman spectroscopy. In that work only weak structural changes were observed in silica glass after electron irradiation. The reason of it is that electrons with high energies have weak interaction with glass and pass through the glass sample with small energy losses. Electrons with $E = 50$ keV lose all their energy in a glass layer of 25–30 μm thick, and the main part of this energy is spend to chemical bands break. That's why, electron beams with relatively low electrons energies are promising for modification of optical properties of optical materials.

CONCLUSIONS

Experiments have shown that for $E = 50$ keV there are three specific intervals of electron irradiation doses, which produce different changes in silica glass structure. In the first one, for doses less, than 10 mC/cm², the destruction of siloxane rings, composing glass network, takes place. In the second one, for doses 10–40 mC/cm², the restoration of siloxane rings after electron irradiation takes place, because of high concentration of structural defects, such as Q^2 and Q^3 . In the third one, for doses more, than 40 mC/cm², the restoration of siloxane rings is not observed. The reason of it is the destruction by electrons of Q^2 , Q^3 and Q^4 structural units, which can compose siloxane rings. The influence on electron irradiation dose on crystalline quartz structure differs considerably from the influence of irradiation dose on structure of silica glass.

FUNDING

This work was financially supported by Russian Science Foundation (Project no. 20-19-00559).

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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