EVALUATION OF IRRADIATION EFFECTS ON SILICA GLASS BY ELECTRON-BEAM USING CATHODOLUMINESCENCE

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It has been reported for geosynchronous spacecrafts that the high density arc plasma short-circuits the adjacent array strings and the solar array supplies the current to the trigger arc that can grow into damaging sustained arc. Since the exposure of the solar panels to high-energy electrons can promote the triggering of the arc, it is important to understand the basic mechanism of charging phenomena in the cover glass of the solar array. We studied irradiation effects of keV-order electron beam irradiation on various types of glasses by means of cathodoluminescence (CL). The CL measurements were performed under electron-beam irradiation using a scanning electron microscope as an electron beam source. The CL spectrum was measured in a monochromatic mode, while the time response of the CL was measured in a panchromatic mode, where the integrated intensity of the whole CL spectrum was studied. Samples were silica glasses containing various concentrations of impurity. Also, various types of silica including thermal oxide, borosilicate glass, and Ge-doped silica were studied for comparison. The evaluation technique using the CL effects on glass can be also applicable to the evaluation of surface discharge for polymers used for spacecrafts.

Introduction

Electrostatic discharges in the solar panels on geosynchronous spacecrafts are a suspected cause of their anomalous operation. The accidents involving the electrostatic discharge in the space environment must be deeply related with an interaction between dielectric materials and high-energy charged particles. The high energy particles include electrons and protons abundant in the space environment, which are produced by unexpected solar flares. However, the detailed mechanism of the damage on the solar panels by the incident high-energy particles has not been well understood.\(^{1-3}\)

Generally, valence electrons of insulators such as glass are excited by the incident high-energy particles from the ground state to the conduction band, as shown in Fig. 1, leading to the formation of electron-hole (e-h) pairs, or weakly bound e-h pairs, so-called excitons. When the recombination of e-h pairs or excitons is accompanied by emission of photons, a luminescence phenomenon can be observed. In particular, the luminescence phenomenon induced by electron beam irradiation is called cathodoluminescence (CL). When the positive or negative charges created by the electron beam irradiation are trapped at the site of defects or impurities, the changes in the CL intensity is expected due to the decreased numbers of the recombination of e-h pairs or excitons. Also, induced defects in insulators by the incident high-energy electrons can be another trapping sites for the charges. Therefore, useful information on the deterioration mechanisms of insulators including charging phenomena under extreme environments like the outer space, can be gained through the CL measurements of insulators under the electron beam irradiation.

The purpose of this study is to evaluate irradiation effects on silica glass by a keV-order electron beam through the measurements of CL spectra and the time response for various types of silica glass with different concentrations of impurities.
Experimental Techniques

We used various silica glasses containing different concentrations of impurities. Also, thermal oxide of silicon, borosilicate glass (Pyrex), cover glass, and slide glass for optical microscope, and Ge-doped silica were studied for comparison. The silica glasses used for the electron beam irradiation are $10 \times 10 \text{mm}^2$ in dimension and 1mm in thickness. Table 1 shows the impurity concentration of the silica glasses.

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<td></td>
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</table>

Table 1. Impurity concentration of silica used in this study (unit: ppm)

Figure 1 shows the schematic illustration of the system for electron-beam irradiation and CL measurements. We measured the CL spectra and intensity of the silica glasses as a function of the irradiation time. We used a conventional scanning electron microscope (SEM, Shimadzu, SSX550) as an excitation source of the CL. The parameters of the SEM operation at the time of the CL measurement are kept (acceleration voltage: 15 kV, emission current: 100 μA, magnification: 500 times, probe size: 7.99 (arb. units), aperture size: 1 mm in diameter).

The CL spectrum was measured with a CL monochromator (GATAN-UK, Mono CL3) that was installed to a port of the vacuum chamber of the SEM. The electron beam was focused at the surface to about 200 nm in diameter and scanned over the area of $150 \mu m \times 200 \mu m$. The CL from the sample surface was collected and dispersed by the monochromator equipped with a photomultiplier. We measured a spectrum of wavelength ranges from 250 nm to 850 nm. The time to need for the measurement then was about 11 minutes. The time response of the CL intensity was measured under a panchromatic mode where the integrated intensity of the whole spectrum from 250 nm to 850 nm was measured by the PMT.

We also estimated the fluence of electrons incident to the sample by measuring a current by a current meter (ADVANTECH, TR8652) outside of the vacuum chamber through a conducting wire out of aluminum foil with a thickness of 12 μm over the glass sample.
Results and discussion

In order to estimate the irradiation effects on silica glass by electron beam, the depth distribution of incident electrons and an energy loss was estimated using a simulation code CASINO\(^{16}\). Figures 3 and 4 show the tracks of electrons with an energy of 15 keV incident to SiO\(_2\) and Al, respectively. As shown in Fig. 3, the penetration depth into aluminum of the electrons is less than 2 \(\mu\)m. Therefore, the aluminum foil with a thickness of 12 \(\mu\)m used is thick enough for the electron current measurement. The measured current under electron beam irradiation was 50 nA. Based on this current, we can estimate electron fluence at the time of the time responses measurement in the following expressions. Using the value of electric current value, irradiation time of 72 minutes over the scanning area of 150 \(\mu\)m \(\times\) 200 \(\mu\)m, the electron fluence is estimated to be \(4.5 \times 10^{18}\) (electron/cm\(^2\)). Since the secondary electron release effect is not taken into account here, it is necessary to use a Faraday cup for the more precise evaluation of the electron fluence.

The penetration depth of the 15 keV electrons into SiO\(_2\) does not reach 3 \(\mu\)m at the maximum. Therefore, the irradiation effects including defect formation and charging are also limited to the surface of the silica glass.

Fig. 3 Results of the CASINO simulation on 15 keV electrons into aluminum.

Fig. 4 Results of the CASINO simulation on 15 keV electrons into SiO\(_2\).

Figures 5 and 6 show, respectively, a CL spectrum and the time response obtained for the thermal oxide on silicon as a standard sample. As for the CL spectrum of thermal oxide, which contains negligibly small impurities compared with silica
glass, a CL peak was observed only at the wavelength of 460 nm. The 460 nm CL band is regarded as the emission of light peculiar to amorphous SiO₂. As previously reported, this band is ascribed to the recombination of the self-trapped excitons of SiO₂\(^5\). The time response curve of the CL in Fig. 6 comprises growth and decay components, followed by a steady state component.

Figures 7 and 8 show CL spectra and the time response for various silica glasses with different concentration of impurities. These CL spectra exhibit the intrinsic 460 nm band, as in the case of thermal oxide, while the intensity varies from sample to sample. Impurity-related CL bands were also observed at 300-400 nm and 650 nm. As already seen in the case of thermal oxide, the time response of the intrinsic CL band at 460 nm comprises growth and decay components. The growth rate of CL increases with the OH content and other impurities. The decay of the CL intensity can be understood in terms of the accumulation of damages such as defect generation in silica glass by 15 keV electrons. Correlation of the CL time response with charging effect of silica glass is discussed in term of the trapping and defect generation in silica glass.

First, we discuss the decay process of the time response of the CL in Fig. 8. The decay of the CL starts 10 to 20 minutes after the electron beam irradiation. Generally, it is thought that electron beam irradiation is accompanied by a lattice distortion and defect generation within glasses. Since these defects can act as the capture centers of electrons or holes, the decay of the CL can be understood in terms of decreased probability of radiative recombination of the self trapped excitons. Therefore, more detailed analysis on the CL curves can provide information on the mechanism of the trapping of e-h pairs or excitons, thereby affecting the charging process of glasses under electron beam irradiation.

Next, we compare the growth process of the CL response curve in detail. When compared with the sample EDH, the rapid growth just after the start of electron beam irradiation is observed in Fig. 8 for samples N and ES, both containing impurities such as OH and Al. Such a rapid CL growth can be explained by the increased radiative recombination times of the excitons or e-h pairs under the existence of impurities. A possible explanation for the increased radiative recombination process is the effect of the increasing temperature during electron beam irradiation. Even if electrons and holes are captured once by impurities and defects, they can be thermally released again. Therefore, the initial growth of the CL can be regarded as a transient phenomenon. We also note for the CL of for the less pure sample N in Figs. 7 and 8, the presence of a spike, which suggests the intermittent emission of light. This signal tends to be easily observed for less pure glasses. The spike can be ascribed to a discharge, which occurred as a result of increased charge accumulation in the less pure silica.

![Fig. 7 CL spectra of various silica glasses.](image1)

![Fig. 8 Time responses of various silica glasses.](image2)
Figures 9 and 10 show the CL spectra and time response of the samples containing impurities such as GeO$_2$ and B$_2$O$_3$. Various peaks were observed depending on impurities and their concentration. The initial growth rate of the PL intensity in Fig. 10 is much higher when compared with the relatively pure silica glasses shown in Fig. 8. Since the time response of the CL is dominated by these impurity related peaks as shown in Fig. 9, such different behavior in the time response can be ascribed to the extrinsic mechanisms of the CL involving impurity-related luminescent centers.

![Figure 9](image1.png)  
**Fig. 9** CL spectra of the samples for comparison, including 7 mol% Ge doped silica, Pyrex SiO$_2$: 80.9 %, B$_2$O$_3$: 12.7 %, Al$_2$O$_3$: 2.3%, Na$_2$O: 4 %, Cover glass: MATSUNAMI, MICRO COVER GLASS, Slide glass: MATSUNAMI, MICRO SLIDE GLASS.

![Figure 10](image2.png)  
**Fig. 10** Time response of the samples, including 7 mol% Ge doped silica.

Summary

We studied electron-beam irradiation effects on various silica-based glasses. We observed an intrinsic luminescence band due to self-trapped excitons in silica glass at 460 nm by the CL measurements. Luminescence bands due to impurities were observed at 300-400 nm and 650 nm. The time response of the CL comprises increase and decay components, where the recombination of the self-trapped excitons is affected by the presence of impurities and by electron-beam-induced defects. In addition, the intermittent emission of light by an electric discharge was observed for silica glass with many impurities.

By the CL measurements, we can monitor the trapping processes of electrons and holes in various types of glasses under electron-beam irradiation, which is a possible cause of the charging of insulators in space. Further study on the CL on insulators such as silica-based glass gives further insight into the charging effects of insulators used in spacecrafts.

References