Cathodoluminescence and electron spin resonance characterization of synthetic quartz

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Cathodoluminescence (CL) and electron spin resonance (ESR) detect structural defects and impurities in quartz. The E_1 ' center can be easily identified by ESR method. In the ESR, the E_1 ' center is defined as an unpaired electron in a single silicon sp³ orbital oriented along a bond direction into an oxygen vacancy (Feigl *et al.*, 1974). However, the assignment of E_1 ' center in CL has been discussed so far in comparison with the results by ESR method (*e.g.*, Stevens-Kalceff, 2009). Usually, natural quartz has various impurity elements and defects. In this study, we have conducted to clarify the E_1 ' center in synthetic quartz by comparison with CL and ESR.

Single crystal of synthetic quartz without any impurities (Miyazaki Epson Co.) was employed for CL and ESR measurements. Sample was sliced perpendicularly to the c axis, and its surface was polished. Electron were irradiated on the surface using a Cockcroft-Walton accelerator located at Takasaki Research Center (JAERI). Implantation energy of electron beam was at 1.0 MeV at 5 stages of 4.51×10^3 -3.20×10^4 J/cm². CL color images were obtained using the Luminoscope (ELM-3). CL spectroscopy was carried out by an SEM-CL system, which consists of an SEM (JEOL, JSM-5410LV) combined with a grating monochromater (Oxford, Mono CL2). All CL spectra were corrected for total instrumental response, which was measured using of a calibrated standard lamp. ESR spectra were obtained by an ESR spectroscopy (JEOL, JES-PX2300).

The ESR spectra obtained from annealing sample at 600 °C exhibit no signal assigned to E_1 ' center at g = 2.001, whereas weak one is recognized in untreated sample. According to Toyoda *et al.*, (1996), the heating up to 600 °C almost quenches the signal of E_1 ' center. The results of the ESR measurements indicate that the signal of E_1 ' center in both annealed and untreated samples pronounces with an increase in the irradiation dose. It clearly shows a formation of E_1 ' center. in quartz by electron irradiation. CL analysis by a spectral deconvolution method reveals five emission components related to defect centers at 1.77 eV, 1.9 eV, 1.95 eV, 2.3 eV, 2.7 eV and 3.14 eV for annealed and untreated samples. An increase in electron-irradiation dose results in an increase in the intensity of the component at 1.77 eV. This result suggests that the emission at 1.77 eV in a red region closely relates to the E_1 ' center determined by ESR method, whereas the emission component at around 2.7 eV has been assigned to the E_1 ' center involved with STE in quartz.

Keywords: quartz, cathodoluminescence, electron spin resonance, E1' center