Changes in Core-Level Photoemission Spectra of Quartz after Heated Up to 1100ºC

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Silicon dioxide (SiO_2), also known as silica, is a compound abundantly present in the Earth's crust. It occurs naturally as quartz and is widely used in applications such as glass and insulating films in semiconductor devices due to its excellent insulating properties. Quartz is known to undergo a structural phase transition at 575ºC. SiO_2 further exhibits polymorphism, with crystalline structures including quartz, cristobalite, and tridymite. Under extreme high-temperature conditions, changes in the electronic structure and crystal structure of SiO_2 may occur, potentially affecting the performance of devices.

The objective of this experiment is to analyze the changes in the state of SiO_2 (quartz) crystals subjected to prolonged exposure to high-temperature environments using photoelectron spectroscopy.

The material used in this experiment was quartz with dimensions of 5×5×0.5 mm and of (0001) orientation. Heating treatment was performed in air as follows. The sample was heated up to 700 ºC linearly in four hours, kept at 700ºC for three hours, heated up to 1100ºC in six hours, kept at 1100ºC for five hours, followed by rapid cooling in air to room temperature.

The XPS measurements were carried out at SA-1 of SR Center in Ritsumeikan University. Two types of X-rays were used: Al Kα and Cr Kα which probe surface and bulk, respectively. Measurements were taken on the quartz sample both before and after heating. The resulting data are shown in Figs. 1 and 2. Let us examine the four Si 2p peaks measured using Al Kα (φ200 μm diameter) and Cr Kα (φ100μm) X-rays before and after heating. Before the heating treatment, the Si 2p binding energy was about the same between Al source and Cr source. After the heating treatment, the binding energies changed. Notably, the shift was larger in the case of the Al source compared to the Cr source. A similar trend was also observed in the O 1s peaks.

For this reason, we can conclude that heating induces noticeable changes in the electronic states of SiO_2, as reflected in the shifts of both the Si 2p and O 1s peaks. These shifts suggest modifications in the chemical environment or potential charging effects on the surface. The greater shift observed with Al Kα excitation compared to Cr Kα may be attributed to differences in probing depth or surface sensitivity between the two X-ray sources.

We should note that the difference in binding between Si 2p and O 1s peaks was basically the same in all conditions even after the heating treatment. Therefore, we consider that the changes in the binding energy may directly reflect the position of the Fermi level (E_F) in the band bap. After the heating treatment, E_F approaches the top of the band gap, in other words, E_F approaches the bottom of the conduction gap. The direction of the E_F shift indicates that the sample became more n-type. This suggests that oxygen vacancies were introduced by the heating treatment.

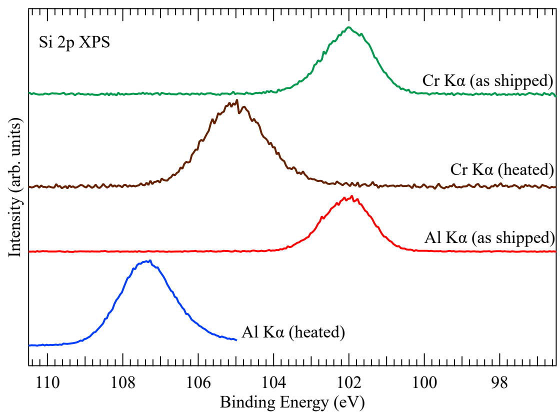

Fig. 1 Si 2p XPS of quartz before and after heating in air.

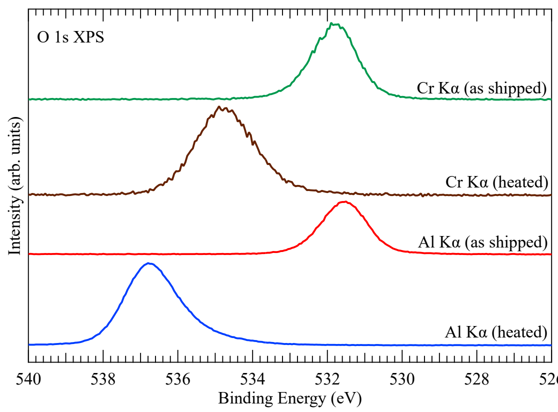

Fig. 2 O 1s XPS of quartz before and after heating in air.