

Chemical state modification during the Si-water reaction

Shugo Nishimura¹, Shiho Fujimura², Kei Mitsuhara¹, Masaru Takizawa¹,
and Eiji Yamasue²

1) Department of Physical Sciences, Faculty of Science and Engineering, Ritsumeikan University, 1-1-1 Noji-Higashi, Kusatsu 525-8577, Japan

2) Department of Mechanical Engineering, Faculty of Science and Engineering, Ritsumeikan University, 1-1-1 Noji-Higashi, Kusatsu 525-8577, Japan

A silicon sludge is a waste discarded during the manufacturing process of Si wafer, and it is often disposed though it includes Si metal [1]. Since Si produces hydrogen by reacting with water, Si sludge may be able to be reused as a substitute resource of Si on hydrogen generation. However, an impurity in Si sludge, such as Al, inhibits the reaction of Si with water [2]. To overcome this problem, the mechanism of the Si-water reaction and the influences of impurities should be addressed. In this study, we carried out X-ray absorption fine structure (XAFS) measurements to directly reveal the chemical state modifications of Si powders for the Si-water reaction.

The XAFS measurements were carried out at BL-13 of SR Center in Ritsumeikan University. The Si *K*-edge XAS spectra were obtained in the total electron yield (TEY) mode. Incident energies were changed by InSb(111) double crystal monochromator. Si powder from Wako Pure Chemical Industries Co., Ltd was used in the experiment. The powder was crushed by using a planetary ball mill with an agate jar and 20 balls for 30 minutes at 200 rpm to reduce the particle size of the powder and increase the surface area. After that, the milled powder was immersed in water to produce hydrogen. The water temperature was kept at 50 °C while stirring. The time to immerse the sample in water was 120 minutes.

Figure 1(a) shows Si *K*-edge XAS spectra before and after the Si-water reaction. There are two peaks in the Si *K*-edge XAS spectra. One is the peak around 1840 eV coming from existence of Si⁰. The other comes from Si oxide (~Si⁴⁺) at about 1847 eV. After the Si-water reaction, Si oxide component increases. Focusing on Si oxide (~Si⁴⁺) at about 1847eV in Si *K*-edge XAS spectra, the peak position slightly shifts after the Si-water reaction [Fig. 1(b)]. Since water contains hydroxide ion, it may affect the Si-water reaction. Comparing the Si *K*-edge XAS spectra of SiO(OH)₂ with SiO₂, the peak positions were slightly different in spite of the same Si⁴⁺. In order to evaluate the amounts of Si-hydroxide and SiO₂, the Si *K*-edge XAS spectra are decomposed with three reference spectra [Si, SiO₂, and SiO(OH)₂], as shown in Fig. 2. The milled powder (before the

Si-water reaction) is found to be mainly composed of Si and SiO₂ [Fig. 2(a)]. After the Si-water reaction, on the other hand, the Si *K*-edge XAS spectrum is well reproduced with the sum spectrum of the Si and SiO(OH)₂, not SiO₂ [Fig. 2(b)]. Thus, the Si-water reaction, in which hydrogen is produced, changes a surface metallic Si to form Si-hydroxide.

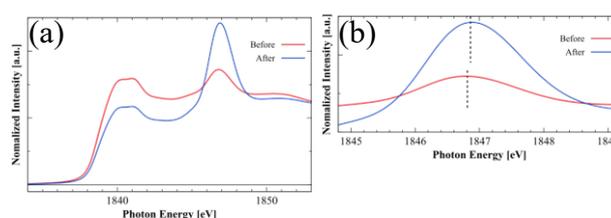


Fig. 1 Si *K*-edge XAFS spectra before and after the Si-water reaction. (a) Wide region. (b) Silicon oxide (~Si⁴⁺) region around 1847eV.

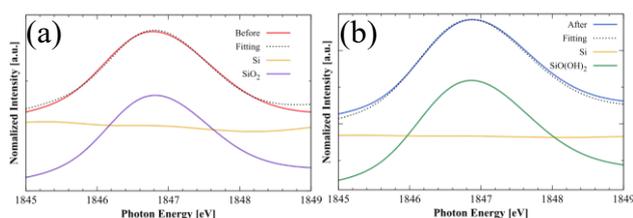


Fig. 2 Decomposed Si *K*-edge XAS spectra around 1847eV. Before (a) and after (b) the Si-water reaction.

References

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