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Quantitation of the Chemical State of Si Powder Milled with Al Powder

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Si sludge is a waste discarded in the manufacturing processes for Si wafer, although it includes Si metal [1]. Si metal can be used for hydrogen production, whose sustainable process has attracted much attention, since Si reacts with water. According to previous study [2], Si sludge and Si powder milled with Al powder contain not only Si metal but also compounds consisting of Si, O and Al, and besides, and they do not react with water. It is important to reveal the amount of the compounds because it may inhibit hydrogen production. In this study, we carried out X-ray absorption fine structure (XAFS) measurements to quantify the chemical state of Si powder milled with Al powder using linear combination fitting (LCF).

The XAFS measurements were carried out at BL-13 of SR Center in Ritsumeikan University. The Si *K*-edge XAFS spectra were obtained in the total electron yield (TEY) mode. The detection depth using TEY mode is about 70 nm for the measurement of Si *K*-edge XAFS measurement [3]. Incident energies were changed by InSb(111) double crystal monochromator. We have measured Si powder, Si+Al2.6wt%, sand, zeolite and cleaned Si powder. Si+Al2.6wt% was made by mixing Si powder and 2.6wt% Al powder using a planetary ball mill with an agate jar and 20 balls for 30 minutes at 200 rpm. Sand is composed of silicon dioxide. Zeolite is mainly composed of aluminosilicate. The clean Si powder was created by etching the Si powder in NH_4F(40%w/w) and $(NH_4)_2SO_3$(1%w/w) for 10 minutes. The surface was removed of oxide and terminated with hydrogen.

Fig. 1 shows Si *K*-edge XAFS spectra for Si powder and the LCF result with cleaned Si powder and sand. One peak around 1840 eV comes from the Si^0 component. The other around 1847 eV comes from Si dioxide (Si^{4+}). However, the spectral feature around 1842 - 1846 eV is not reproduced by LCF. This feature is considered to come from intermediate Si oxides, which exist in Si powder. Therefore, we have to consider this component (this residual is named "other" in the following).

Fig. 2 shows Si *K*-edge XAS spectra for Si+Al2.6wt% and the LCF result with cleaned Si powder, sand, zeolite and other. The respective percentages of each component are shown in Table 1. In Fig. 2, fitting result is in good agreement with Si+Al2.6wt%. The result indicates that the ball milling of Si and Al contains not only Si oxide but also 5% aluminosilicate.

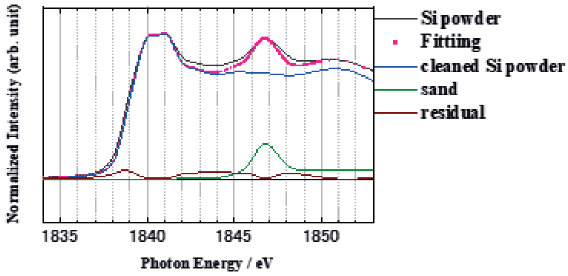

Fig. 1 Si *K*-edge XAS spectra for Si powder and the LCF result.

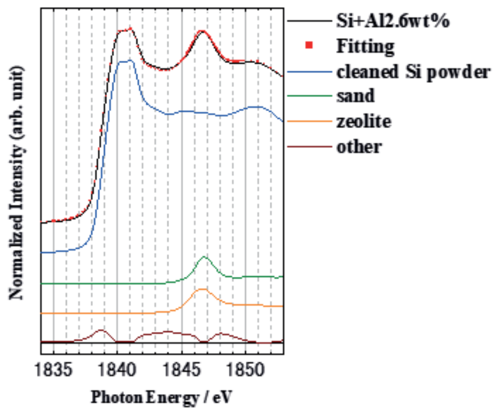

Fig. 2 Si *K*-edge XAS spectra for Si+Al2.6wt% and the LCF result.

Table 1 Chemical composition of Si+Al2.6wt%

	cleaned Si(%)	sand(%)	zeolite(%)	other(%)
Si+Al2.6wt%	84	4.4	5.0	6.6

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