EXAFS analysis of PdTe catalyst

Kiyotaka ASAKURA¹, Wang-Jae Chun¹ ¹², Yuichiro KOIKE³, Norikazu KONISHI⁴, Hideyuki TAKAHASHI⁵, Atsushi MURAMATSU⁴, Nobuhiro OHNO⁶
¹Catalysis Research Center, Hokkaido University,
²CREST-JST
³KEK-PF, Tsukuba, Ibaraki 305-0801, Japan
⁴Institute of Multidisciplinary Research for Advanced Material, Tohoku University, Katahira, Sendai 980-8577, Japan
⁵Graduate School of Environmental Studies, Tohoku University, Aoba, Sendai, 980-8579, Japan
⁶Mitsubishi Chemical Company, Kurashiki, 712-8054, Japan

Introduction

PdTe bimetallic systems show high activity for oxidative diacetoxylation reaction of butadiene. Liquid phase reduction method gives PdTe nanoparticles with homogeneous composition and size.[1] However, the inner structure of PdTe bimetallic nanoparticle is still unknown. We have applied EXAFS spectra to determine the structure of PdTe bimetallic nanoparticles.

Experimental

The EXAFS measurements were carried out at NW10A of PF-AR, with 6 GeV – 50 mA. The X-rays were monochromatized with a Si(311) double crystal monochromator. The monochromatized X-ray was focused to the sample by a Pt-Coated bent cylindrical mirror. The critical energy for the total reflection of the mirror was set at 42 keV. The I₀ and I signals were measured by Ar filled ionization counters. The PdTe nanoparticles were prepared by the liquid reduction method. SiO₂ was impregnated with a mixture solution of PdCl₄/EDTA/TeCl₂/citric acid and hydrazine, followed by a reduction treatment.

Results and discussion

Figure 1 shows Pd and Te EXAFS oscillations together with Pd20Te7 alloy powder. In the Pd 20Te7, Pd was surrounded by both Pd and Te in the first nearest shell while Te was only surrounded by Pd. The Figure 1 also shows the PdTe deposited on SiO₂. The EXAFS oscillations on both edges are quite similar to those Pd20Te7, indicating the formation of Pd20Te7 small particles. Closely looking at Pd K-edge EXAFS in high k-region in PdTe nanoparticle on SiO₂, one may find a larger EXAFS oscillation than that in Pd20Te7 where EXAFS oscillation damps around k=130 nm⁻¹. We postulated the presence of 10 % Pd species and simulated the EXAFS data. The synthesized data of Pd20Te7+Pd agree well with that of the observed Pd K-edge EXAFS of PdTe nanoparticles. The result indicated that the nanoparticle might be a mixture of Pd20Te7 bimetallic and Pd metallic states.