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EXAFS analysis of PdTe catalyst

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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 nanopartiles.

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 I_0 and I signals were measured by Ar filled ionization counters. The PdTe nanoparticles were prepared by the liquid reduction method. SiO_2 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 SiO_2. 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 SiO_2 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.

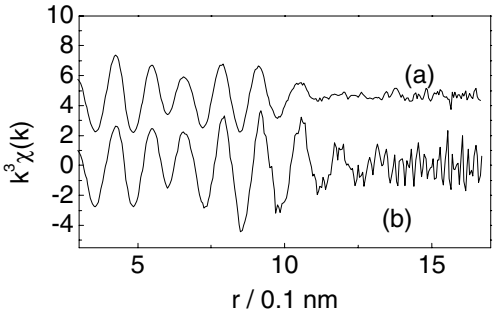

Figure 1 Pd Kedge of EXAFS oscillations of Pd20Te7 (a) and PdTe/SiO_2 (b).

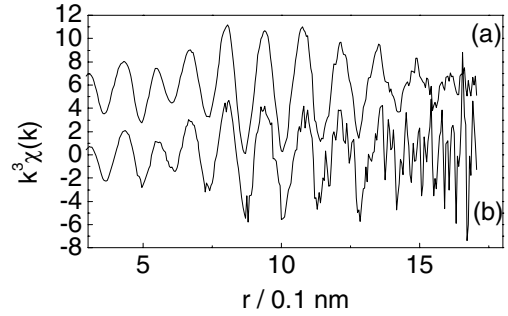

Figure 2 Te K-edge of EXAFS oscillations of Pd20Te7 (a) and PdTe/SiO_2 (b).

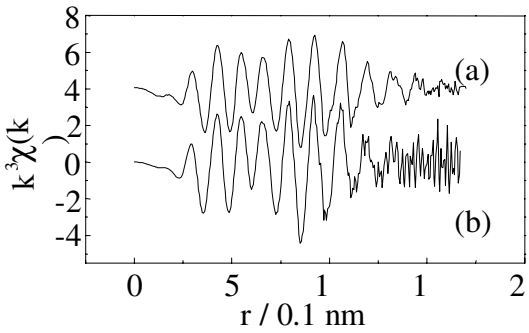

Figure 3 synthesized Pd K-edge EXAFS oscillation from the EXAFS oscillations of Pd20Te7 and Pd (ratio is 0.9:0.1)(a) and observed data of PdTe/SiO_2 (b)