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EXAFS analysis of the Rh-Te alloy nanoparticles prepared by liquid phase reduction method

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Introduction

The rhodium tellurium (RhTe) bimetallic nanoparticle works as the catalyst of oxidative diacetoxylation reaction of butadiene. In our previous study, RhTe nanoparticles were prepared by liquid-phase reduction in the presence of chelating agents. In this study, we have applied the liquid phase reduction method to prepare RhTe nanoparticles in organic solvent. In this paper, we have studied the structure of prepared Rh-Te nanoparticles by using EXAFS analysis to figure out preparation mechanism.

Experimental

The EXAFS measurements were carried out at BL14B2 of SPring-8 with 8 GeV - 99.5 mA (Proposal No. 2011A1973) and at NW10A of PF-AR with 6.5 GeV – 60 mA. The X-rays at BL14B2 passed through a Si(311) double-crystal monochromator and focused onto the sample. The Rh K-edge EXAFS data was collected in a transmission mode using I_0 and I ionization counters filled with 100% Ar and 30% Kr/Ar, respectively. The Te K-edge EXAFS data was collected in a transmission mode using I_0 and I ionization counters filled 20% Kr/Ar and 100% Kr for Te K-edge, respectively. The X-rays at NW10A 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 and Kr filled ionization counters, respectively. The RhTe nanoparticles were prepared by the liquid-phase reduction method using ethylene glycol.

Results and discussion

Figure 1 shows the Rh K-edge FT-EXAFS spectra of RhTe alloy nanoparticles on SiO_2 support, RhTe alloy nanoparticles powder without support, reference of RhTe alloy bulk and Rh foil. The peak around 0.2 - 0.3 nm corresponds to the Rh-Rh or Rh-Te distance and the peak of the reference of Rh_3Te_2 ingot. Rh metal might not be formed, since there was not peak at 0.4 - 0.5 nm corresponding to Rh-Rh bond derived from fcc Rh.

Figure 2 shows the Te K-edge FT-EXAFS spectra of RhTe alloy nanoparticles on SiO_2 support, RhTe alloy nanoparticles powder without support, the reference of RhTe alloy bulk and Te powder. The peak around 0.2 - 0.3 nm corresponded to the Te-Rh distance. In the case of RhTe without support, the peak around 0.3 - 0.4 nm corresponding to the peak of Rh_3Te_2 was observed.

These results show that the resulting nanoparticles might be composed of Rh-Te without Rh-Rh phase. In addition, Rh_3Te_2 alloy nanoparticles were synthesized when the nanoparticles were prepared without SiO_2 support. The preparation method might affect the formation of Rh-Te bond by uniform exist of Rh and Te precursors in reaction solution.

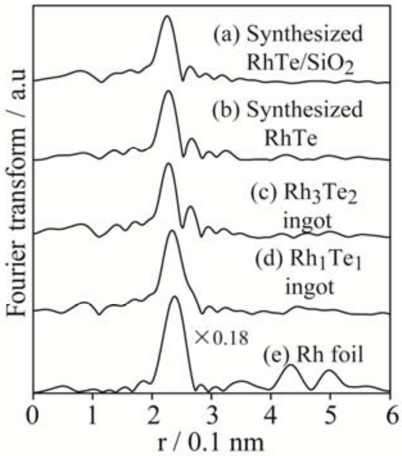

Figure 1. Rh K-edge FT-EXAFS spectra of (a) RhTe nanoparticles on SiO_2, (b) RhTe nanoparticles, (c) Rh_3Te_2 ingot, (d) Rh_1Te_1 ingot and (e) Rh foil

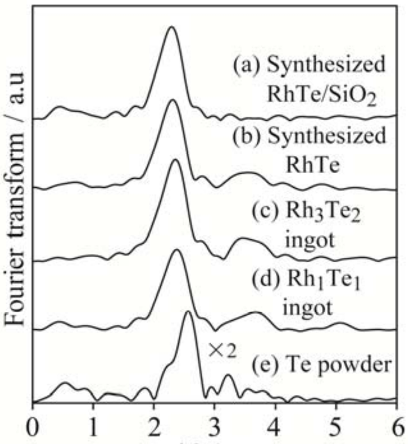

Figure 2. Te K-edge FT-EXAFS spectra of (a) RhTe nanoparticles on SiO_2, (b) RhTe nanoparticles, (c) Rh_3Te_2 ingot, (d) Rh_1Te_1 ingot and (e) Te powder