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# Structural Analysis of Bimetallic Nanoparticles prepared in Sub- and Supercritical Fluids by means of EXAFS

Yoshifumi KIMURA*[1], Masaki UEJI[2], and Masafumi HARADA[3]
[1]International Innovation Center, Kyoto University, Kyoto 606-8501, Japan
[2]Department of Chemistry, Graduate School of Science,
Kyoto University, Kyoto 606-8502, Japan
[3]Department of Textile and Apparel Science, Faculty of Human Life and Environment,
Nara Women's University, Nara 630-8506, Japan

## Introduction

Bimetallic nanoparticles nowadays attract so much attention of a lot of chemists as sources of nanomaterials and their synthetic methods have been explored extensively. In our group we have developed a new synthetic method of making bimetallic nanoparticles composed of noble metals by using high-temperature and high-pressure fluids[1]. We have succeeded in making a Pt/Rh bimetallic clusters using the high-temperature and high-pressure water/ethanol mixture solution under the existence of a protective polymer (poly(N-vinyl-2-pyrrolidone), PVP)[2]. The structure of bimetallic cluster was quite different from that is made under the ambient condition. In this report, we will present the results of the synthesis of Pt/Pd and Au/Rh clusters.

## Experimental

Platinum/palladium (Pt/Pd) bimetallic nanoparticles were synthesized by passing mixture solution of $H_2PtCl_6 \cdot 6H_2O$, $PdCl_2$ and PVP through the high-temperature and high-pressure SUS cell[1]. In the case of gold/rhodium (Au/Rh) solutions, we used $NaAuCl_4 \cdot 2H_2O$ and $RhCl_3 \cdot 3H_2O$. The temperatures and pressure were 473-573 K and 25 MPa, respectively. As solvents, we used water ethanol, and their 1:1 mixture. The obtained samples were condensed and then poured into cells for EXAFS measurements, and Pt-$L_{III}$, Au-$L_{III}$, Pd-K and Rh-K edges' EXAFS spectra were collected at room temperature in a transmission mode at BL-10B, -7C and -9A to estimate the coordination numbers (N's).

## Results and Discussion

Figure 1 shows typical examples of EXAFS spectra for the colloidal dispersions of Pt/Pd nanoparticles. As is shown in the figure, the nanoparticle is alloyed. The results of the analysis are shown in Table 1. In both edges, the coordination numbers are relatively larger than those made for the ambient condition and the values are similar for both metals. This suggests that the particle is relatively large and randomly alloyed. On the other hand, in the case of Au/Rh, the result of the EXAFS suggests that the metallic particle was not alloyed.

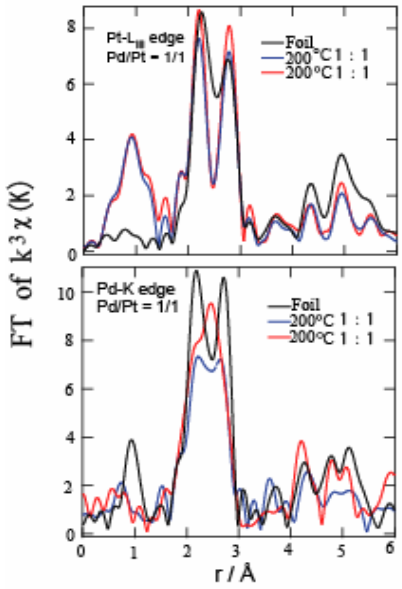


Fig. 1 Fourier transforms of EXAFS spectra for Pt-$L_{III}$ edge and Pd-K edge of Pt/Pd colloidal dispersions produced at different temperatures in comparison with the bimetallic alloy foil (Pt/Pd=1/1).

Table 1. Results of the analysis of the EXAFS for the Pt/Pd colloidal dispersions.

| Absorption edge | Scattering atom | N | |
|---|---|---|---|
| | | 100°C (ambient) | 200 °C (25 MPa) |
| Pd-K | Pd | 2.3 | 5.5 |
| | Pt | 3.0 | 4.5 |
| Pt-$L_{III}$ | Pt | 4.8 | 5.9 |
| | Pd | 1.3 | 4.6 |

## References

[1] Y. Kimura et al., *Colloid and Surface A. Physicochem. Eng. Aspects*, **231**, 131 (2003).
[2] Y. Kimura et al., *Photon Factory Activity Report 2002*, users' report 192.

*ykimura@iic.kyoto-u.ac.jp