

Development of a compact Low-Temperature Polarization-dependent Total Reflection Fluorescence XAFS measurement system using Liquid Nitrogen as a cold agent

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1 Introduction

Metal-support interaction is an essential factor governing the catalytic activity in heterogeneous catalysts. Unveiling the interaction, XAFS could be the most practical tool though it gives only one-dimensional information in general. Overcoming the limitation, Polarization-dependent Total Reflection Fluorescence XAFS (PTRF-XAFS) has provided three-dimensional information that allows determining the metal-support interfacial bonds.^[1] However, this state-of-art technique is still in scope for improvement in S/N ratios.

As an approach, we have proposed a low-temperature PTRF-XAFS measurement. In the previous activity report, we demonstrated a compact LT-PTRF-XAFS system cooled by a liquid N₂, which achieves low cost while satisfying the improvement in S/N ratios.^[2]

This report demonstrates our recent studies in highly dispersed Au and Pt nanoparticles (NPs) on a single crystal TiO₂(110) surface.

2 Experiment

XAFS measurements were performed at the BL-12C of KEK-PF-IMSS (2.5 GeV, 450 mA, Si(111)) with the LT-PTRF-XAFS measurement system^[2] (Fig. 1). Au or Pt L α Fluorescence signals were detected with 7-elements Silicon Drift Detector (XSD50-07, Techno AP, Japan).

Au NPs on TiO₂(110) were prepared by drop-casting with Au colloids (~5 nm, TANAKA, Japan). Pt NPs on the surface were fabricated using a sputtering method (Pt foil, 10 sec., 1400 V, 4 mA, Eiko Eng. Co. Ltd. Japan).

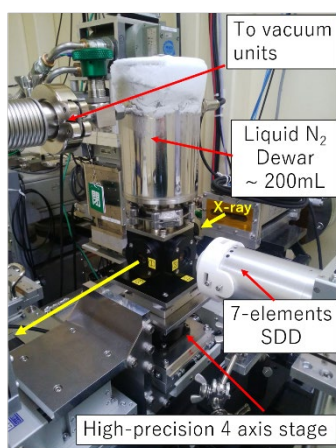


Fig.1 A liquid N₂ cooled LT-PTRF-XAFS system.

3 Results and Discussion

Fig. 2 shows EXAFS spectra of Au NPs on TiO₂(110) at RT and 80 K. The $k^3\chi(k)$ spectra and FT results showed dramatic S/N improvement in the high k -vector region.

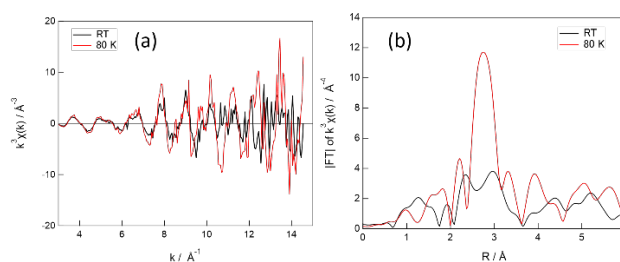


Fig. 2 Au L₃ EXAFS results of AuNPs/TiO₂(110) ($E//[110]$)(a) $k^3\chi(k)$ spectra,(b)FT results $k = 3\sim 14\text{\AA}^{-1}$.

Fig. 3 shows the XANES spectra of Pt NPs on TiO₂(110) where $E//[001]$ (s -pol) and $E//[110]$ (p -pol) to the surface at 80 K. Firstly, We found no specific difference in surface orientations.

Secondly, the XANES features suggested that the electronic state of the Pt NPs was much closer to that of a Pt oxide than a Pt metal. Moreover, preliminary EXAFS analysis proposed that most Pt NPs are oxidized, but small metal particles are also present.

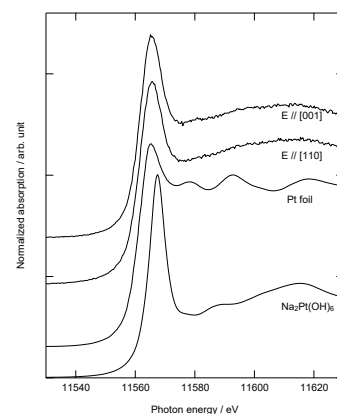


Fig. 3 the LT-PTRF-XANES of Pt NPs on TiO₂(110)

Acknowledgment

This work was supported by JSPS KAKENHI (Grant Number JP25410074). We also appreciated Dr. T. Ano and S. Tsubaki (Tokyo Institute of Technology, Japan) for preparing Pt nanoparticles on TiO₂(110).

References

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