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Development of A Facile Low-temperature Polarization-dependent Total Reflection Fluorescence XAFS (LT-PTRF-XAFS)

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1 Introduction

Metal-support interaction is an important governing factor of 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. To overcome 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 scope for improvement in S/N ratios.

In the previous proposal (2016G551), we have improved the S/N ratios in higher frequency EXAFS oscillations by the first low-temperature PTRF-XAFS (LT-PTRF-XAFS) measurement system with a liquid He-flow cryostat[2]. However, there are issues in the cost of a liquid He and the size of the system. In this report, we introduce our recent progress in a simple LT-PTRF-XAFS system, which achieves low cost while satisfying the improvement in S/N ratios.

2 Experiment

To maintain a total reflection condition at low temperatures, we fabricated a compact liquid N_2 dewar which has no mechanical vibration while reducing the temperature. The modified dewar (*ca.* 200 mL) has an Al shroud with three Kapton window ports. The dewar was installed to a high precision 3-axis stage (Fig. 1).

LT-PTRF-XAFS measurements were performed at the BL-9C/12C of PF (2.5 GeV, 450 mA, Si(111)). Cu Kα Fluorescence signals were detected with 7-elements Silicon Drift Detector (XSDD50-07, Techno AP, Japan). The measurements were carried out in the parallel(s-pol) or perpendicular (p-pol) orientation relative to the electric vector (***E***) of the incident X-rays.

The Cu-HKUST-1 thin film on a single crystal TiO_2(110) was prepared as a sample. Details can be found from elsewhere.[3]

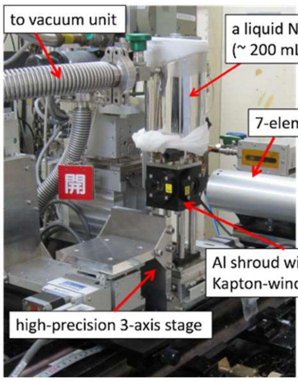

Fig.1 A liquid N_2 cooled LT-PTRF-XAFS system.

3 Results and Discussion

Fig. 2 shows cooling performance. The modified dewar requires only 15 min to 80 K and can keep the temperature for 2 hr with 200 mL $N_2(l)$.

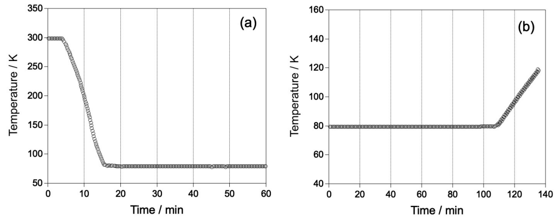

Fig. 2 Performance of the LT-PTRF-XAFS system. (a) required time to 80 K, (b) retention time at 80 K.

Fig. 3 shows the EXAFS spectra of Cu-HKUST-1/TiO_2(110) where ***E***// [001] (s-pol) and ***E***// [110] (p-pol) to the surface at 80 K. We obtained the reasonable polarization-dependent EXAFS spectra at the 80 K, successfully. We also found no specific difference in surface orientations. It suggests the thin film has weak orientation-dependence.

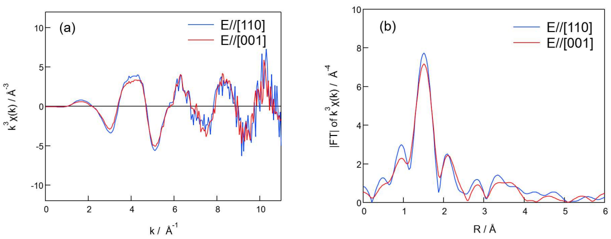

Fig. 3 the LT-PTRF-XAFS of Cu-HKUST-1/TiO_2(110) (a) $k^3\chi(k)$ spectra, (b) FT results ($k = 3\sim11 Å^{-1}$).

The further demonstration will explore various metal nanoparticles highly dispersed on solid substrates.

Acknowledgment

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References

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