Possibility of BCLA+PTRF-EXAFS

Bapurao Bharate\textsuperscript{a}, Yuki Wakisaka\textsuperscript{a}, Daiki Kido\textsuperscript{a}, Takahiro Wada\textsuperscript{b}, Qiuyi Yuan\textsuperscript{a}, Yasuo Takeichi\textsuperscript{i}, Satoru Takakusagi\textsuperscript{a} and Kiyotaka Asakura\textsuperscript{a*}

\textsuperscript{a} Institute for Catalysis, Hokkaido University, Kita 21-10, Sapporo, Hokkaido, 001-0021, Japan  
\textsuperscript{b} Tokyo Medical and Dental University, Yushima 1-5-45, Bunkyo-ku, Tokyo, 113-8549, Japan  
\textsuperscript{c} Institute of Materials Structure Science, High Energy Accelerator Research Organization, Oho1-1, Tsukuba, 305-0801, Japan

A bent crystal Laue analyser combined with polarization-dependent total reflection fluorescence X-ray absorption Fine Structure (PTRF-XAFS) is a promising technique for in situ surface analysis of highly dispersed systems even in the presence of solution.

1 Introduction

Polarisation-dependent total reflection fluorescence extended X-ray absorption fine structure (PTRF-EXAFS) spectroscopy is a powerful technique to determine the three-dimensional structures of metal atoms \((10^{13–15} \text{ cm}^{-2})\) dispersed on atomically flat surfaces\cite{1}. When it is applied to the electrode surface which is covered with solutions, the elastic X-ray scattering of the liquid overlayer becomes large and seriously increases the background X-rays. We used BCLAn(Bent Crystal Laue Analyzer) to remove the elastic scattering from the solution and to allow the PTRF-XAFS measurements under the solutions.

2 Experimental

A thin Pt layer was deposited on a 60-nm-thick polycrystalline Au thin film evaporated on a 10-mm \(\times\) 20-mm Si(100) wafer by self-terminating electrodeposition\cite{3}. Pt was electrochemically deposited on a Au polycrystal from 3 mmol K\textsubscript{2}PtCl\textsubscript{4}–NaCl at pH = 4 with an applied voltage of \(-0.7\) V vs. Ag/AgCl. The deposition time was 20 s. Hereafter, the sample is called Pt/Au/Si. The EXAFS measurements were carried out under the total reflection conditions at beamline BL-15A1. The BCLA was set between the sample and the detector.

3 Results and Discussion

Figure 1 shows the\(\chi(k)\) of L\(_3\)-edge EXAFS of Pt/Au/Si under the total reflection conditions (s-polarization) in a fluorescence mode with and without the BCLA, respectively. In the EXAFS spectrum without the BCLA, strong Au L\(_3\) X-ray fluorescence appeared above 11900 eV (the Au L\(_3\) edge). Even under the total reflection conditions, X-rays could penetrate the bulk and excite the Au edge \cite{2}. The BCLA reduced the Pt\(_L\alpha\) fluorescence X-ray signal to 1/160 of that without the BCLA, i.e., 0.0024 and 0.38 at 11800 eV with and without the BCLA, respectively. However, S/B ratio was tremendously improved to 11000(with BCLA) from 140 (without BCLA) . The S/N ratio of \(\chi(k)\) was 1.5 times better with than without the BCLA. This work suggests that, we can measure an EXAFS of the thick solution covered surface.

References

\[1\] S. Takakusagi, et. al., Top Catal 2013, 56, 1-11.  

* askr@cat.hokudai.ac.jp