Abstract #921, 224th ECS Meeting, © 2013 The Electrochemical Society

Ambient-Pressure “Tender” X-Ray Photoelectron Spectroscopy (APXPS) for *in situ* Study of Liquid-Solid Interface of Pt foil in 6 M KF

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We report on the construction of new ambient pressure X-ray photoelectron spectroscopy (APXPS) endstation capable of operating at 110 Torr and at a “tender” X-ray synchrotron beamline. This new APXPS system is used to characterize the solid-liquid interface region. The new APXPS system is located at the bending magnet beamline 9.3.1 in Advanced Light Source. This beamline operates at photon energies of 2.5 keV to 5.5 keV, which enables us to investigate samples at higher photoelectron probing depths (~ 10 nm to 30 nm) compared to soft X-ray source (~ 1 nm). With this energy range, solid-liquid interface region of a thin-film of liquid on a solid surface can be analyzed. This new APXPS system is utilized for the *in situ* XPS study of electrochemical processes, such as the change of the surface and interface chemical species and electronic properties simultaneously at both the electrode and electrolyte interface, in other words, the electrical double layer region. The electrical double layer is known as the essence of electrochemistry where all the electrochemical reactions take place.[1, 2]

In this study, we focus on studying electrical double layer region using APXPS as an *in situ* technique. Previous studies by Kolb et al. [3, 4] has shown the use of XPS to study *ex situ* the electrical double layer of salt electrolyte on electrode surfaces emersed from the electrolyte. In their study they showed the electrical double layer is preserved after the emersion of the electrode from the electrolyte.[3, 4] In this work, we will show the result from the *in situ* observation of Pt foil in 6 M KF electrolyte using XPS at different holding potentials. The chemical composition of the electrical double layer and the surface characterization of the electrode, Pt, of this system are successfully analyzed *in situ* using the APXPS.

Figure 1 shows the cyclic voltammetry measurement of Pt foil in 6 M KF salt in water from a three-electrode cell system with Ag/AgCl as the reference electrode and another Pt foil as the counter electrode. The whole cell setup is operated inside the APXPS chamber under 16 Torr of water vapor pressure to allow simultaneous XPS measurement of the electrode while maintaining the thin layer electrolyte film formed on the Pt surface. From Figure 1, typical polycrystalline Pt CV plot is observed. The XPS measurements were then taken while holding the electrode at different potentials. Figure 2 shows XPS spectra of O 1s, Pt 4f, and K 2pfrom the electrode with thin electrolyte film present while holding the potential at -0.4 V. It is shown that both the electrode and electrolyte layer is observed using the APXPS while holding the potential of the electrode that is still in contact with the electrical double layer.

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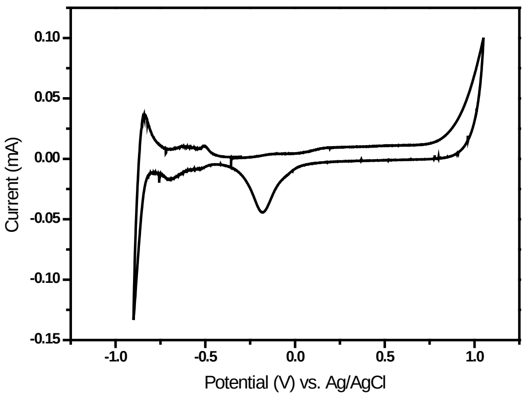

Figure 1. Cyclic voltammetry of Pt foil in 6 M KF in APXPS chamber at 16 Torr of water vapor pressure.

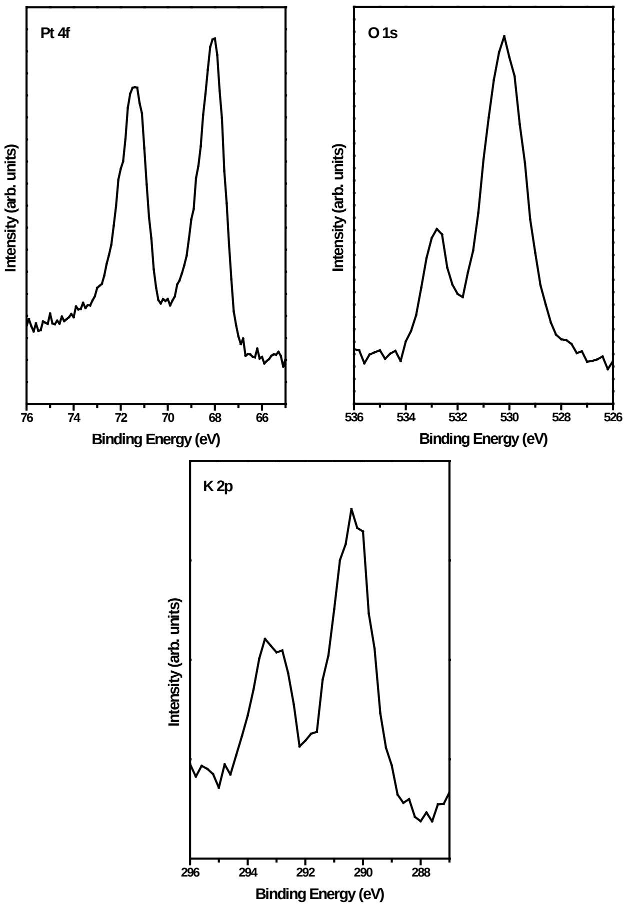

Figure 2: Pt 4f, O 1s, K 2p of Pt foil in 6 M KF measured while Pt foil is in contact with the electrolyte and potential is held at -0.4 V. This shows that both the electrolyte and electrode substrate are observed by “tender” X-ray APXPS technique.

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The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231. S. A. and E. J. C. would like to thank the support of ALS Postdoctoral Fellowship.