Ambient Pressure Photoelectron Spectromicroscopy at Advanced Light Source

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The newly designed ambient pressure x-ray photoelectron spectroscopy (AP-XPS) endstations at BL 9.3.2 and BL 11.0.2, based on differentially pumped electron energy analyzers, have broken the vacuum barrier for soft x-ray photoelectron spectroscopy. Many high profile publications have been generated and it has been recognized by scientific communities beyond the ALS as an important in-situ tool to study water, environmental science, catalysis and many other fields. There is perhaps no better evidence than the fact that eight new AP-XPS endstations are currently under planning or development at US and international synchrotron light sources.

I will give an overview of science projects at BL9.3.2 in heterogeneous catalysis and fuel cell (figure 1). I will also present results from the newest AP-XPS station that we are commissioning. This new instrument, Scienta HiPP4000, is a result of collaboration between ALS and its industrial partner VG-Scienta. Beside an improved performance of electron transmission, this instrument can be operated in spectromicroscopy mode with a spatial resolution of $\sim 20 \,\mu\text{m}$ in one dimension, or angle-resolved mode with 0.5° resolution and a window of $\pm 12^{\circ}$ (Figure 2). When it is operated in maximum transmission mode, the electron detection efficiency is more than fifty times better than the previous endstation at beamline 9.3.2. Spectra have been recorded above 1 Torr and the pumping system has been tested to 6 Torr.



Figure 1. (a) In this study, we used AP-XPS and high pressure scanning tunneling microscopy (HP-STM) to investigate oxidation of Pt(110) as a function of O_2 pressure, temperature and gas composition (CO, O_2). AP-XPS results demonstrate that two different surface oxygen species form on Pt(110) surface at > 500mTorr O_2 : chemisorbed oxygen and island structured surface oxide. HP-STM images under the same conditions confirm the formation of these oxide islands. (b). Using AP-XPS, we can investigate in-situ the solid oxide fuel cell in its operational condition (700C, 1torr of H₂+H₂O) and gain a microscopic and chemically-specific understanding of electrochemical interfaces, especially the electrode-electrolyte-gas three phase boundary.



Figure 2. (a). Angular resolved photoelectron spectrum of graphite in an atmosphere of 200 mTorr CO. In this spectrum, the graphite's band structure and Γ , M, and K points are clearly visible and the non-dispersive CO gas lines are clearly visible as well. (b). Detector image of the Au 4f region and Si 2p region of a 100 μ m Au bar on SiO₂ sample shown in the optical micrograph. The image can be integrated across the *y* dispersion direction to yield XPS spectra as shown or across the energy dispersion direction to reveal spatial heterogeneity.