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## Investigation of the Catalysts' Surface evolution during Electrochemical Reaction under Different pH Environments by Ambient Pressure XPS at NSRRC

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The chemical states of the catalyst surface play vital roles in the various kinds of catalytic re-actions, such as oxygen evolution reaction, oxygen reduction reaction and nitrogen reduction reaction, which will dominate the active sites and the reaction dynamic. Numerous research efforts have been devoted to revealing the active species on the catalyst surface to design the catalyst with great performance. Most innovative spectroscopy techniques are developed and applied to better understand the real-time changes during catalytic reactions nowadays. Several different electrochemical cell designs combined with an ambient pressure X-ray photoelectron spectroscopy (APXPS) end-station have been reported to study the chemical state of the catalyst surface during the electro-chemical reaction. Here, we report an electrochemical cell system inspired by the original design from the BESSY II ISIS beamline coupled with the APXPS endstation at the BL 24A of NSRRC to study the surface evolution of the catalyst under different pH environments during electrochemical reactions.[1, 2] In acidic environment, nafion electrodes with a low-loading amount of Pt catalyst are prepared by the e-gun deposition method to study the species evolution during a redox reaction. The ratio of divalent species on Pt catalyst surface gradually increases during the whole anodic process. In contrast, the tetravalent species appears as the applied potential exceeds the threshold voltage of OER. While stepwise oxidation is observed during the anodic polarization, the hysteresis of oxide species is found on the surface with the incomplete reduction of divalent species, which could be due to the heavier surface oxidation from higher anodic polarization. Additionally, in-operando measurement of valence band spectra shows the decline in electron density near Fermi energy, suggesting the importance of a slightly oxidized surface on the Pt OER catalyst. In an alkaline environment, the zinc substitution iron cobaltite-based spinel structure catalysts ( $Zn_xFe_{1-x}Co_2O_4$ ) are prepared onto the anion exchange membrane (AEM) to study species evolution during the OER reaction. Our results help propose the important intermediate,  $Co^{4+}$  and superoxide species, during oxygen production. More detailed results will be presented in this meeting.

Keywords : Electrochemical Ambient Pressure X-ray Photoelectron Spectroscopy (EC-APXPS), Oxygen Evolution Reaction (OER), Platinum catalyst, Anion Exchange Membrane, Nafion Membrane.

References:

- [1] R. Arrigo, M. Hävecker, M. E. Schuster, C. Ranjan, E. Stotz, A. Knop-Gericke, R. Schlögl, *Angew. Chem. Int. Ed.* 2013, 52 (44), 11660-11664.
- [2] R. Mom, L. Frevel, J.-J. Velasco-Vélez, M. Plodinec, A. Knop-Gericke, R. Schlögl, *J. Am. Chem. Soc.* 2019, 141 (16), 6537-6544.

if "Other", please specify:

I apply for a travel grant

Yes

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