9th Annual Ambient Pressure X-ray Photoelectron Spectroscopy Workhop



Contribution ID: 52

Type: Invited/plenary talk

INVITED: Lab-based APXPS with multimodal IRRAS & ETEM for metal oxide studies

Wednesday, 7 December 2022 10:30 (30 minutes)

With a rise in the number of lab-based APXPS systems, these instruments afford an opportunity to continue the development of multimodal capabilities for more comprehensive information of reactions at surfaces. I will discuss the methods of obtaining multimodal data from infrared reflection absorption spectroscopy (IRRAS) and environmental transmission electron microscopy (ETEM) under the same reaction environments as the lab-based APXPS system at the Center for Functional Nanomaterials at Brookhaven National Laboratory. In situ polarization-dependent IRRAS measurements have been used to confirm the reaction of CO with a Cu₂(111) surface to form CO2. The uncommon IRRAS measurements of a single crystal transition metal oxide surface allow for insights into the geometry of the adsorbates. The combination of APXPS and IRRAS determine that a C 1s binding energy commonly assigned to carbonates is actually CO₂. This study has implications for catalysis and also metal oxide XPS studies, where in this case the adsorbed CO and CO₂ have binding energies higher than other systems. While IRRAS provides more insight into chemical environments on surfaces, ETEM can offer complementary structural information. A 50 micrometer heater on a Nano-Chip used in ETEM was adapted to a gas cell for APXPS measurements. Proof-of-concept measurements show that the heater functions identical to ETEM experiments. The gas lines in the cell enable locally high pressures above the heater, estimated to be 1 mbar with the potential for higher pressures. The rapid temperature increase of the microheater (≤ 1 s) also enables time resolved measurements. The reduction of an oxidized Pd film was followed with 500 ms resolution of the Pd 3d_{5/2} core level. This timescale matches the timescale of ETEM measurements (≥10 ms) of identical processes. Using this Nano-Chip in APXPS offers chemical information complementary to structural changes seen in ETEM. The rapid heating enables new opportunities in time-resolved APXPS. Overall, both the ETEM heater and IRRAS offer ways of combining additional information to yield a deeper understanding of surface reactions beyond the metal oxide chemistry demonstrated here.

if "Other", please specify:

I apply for a travel grant

No

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