

Consortium for Operando and Advanced Catalyst V Characterization via Electronic Spectroscopy and Structure

Co-ACCESS at Stanford Synchrotron Radiation Lightsource (SSRL) at SLAC National Accelerator Laboratory is up and running! This is the first of our semi-annual updates regarding our capabilities and plans. We have initially focused on gasphase heterogeneous catalysis, and providing the capabilities for in-situ/operando catalyst characterization at SSRL.

Introducing Co-ACCESS to those who are not familiar with us, our goals include:

- Provide all levels of assistance to catalysis scientists conducting synchrotroncharacterization experiments, in the framework of collaboration, to maximize the success of their research.
- Develop a suite of *in-situ/operando* reactors to cover a wide range of catalytic chemistry: thermal heterogeneous catalysis (both gas and liquid phase), homogeneous catalysis, and electro-catalysis that are compatible with the various beam lines at SSRL.
- Develop advanced experimental methodologies, e.g. modulation-excitation spectroscopy to differentiate active and spectator species, and combined X-ray absorption/diffraction experiments.
- Develop a multi-modal spectroscopy platform, incorporating FTIR and Raman (and potentially UV-vis) spectroscopies.

Improve beamtime scheduling to accommodate experiments that require prolonged run times (e.g. long-term deactivation studies). Offer guidance and support in all aspects of a synchrotron experiment – from proposal writing, to experimental planning, to data analysis and publication. This will lead to increased efficiency of operations.

- Offer educational opportunities to graduate students to learn advanced data modeling of XAFS data.
- Leverage the user-friendly "can do" attitude of SSRL to focus on demanding experiments that require expertise from diverse backgrounds, and patience.





FTIR Spectrometer

Highlighting our new Nicolet iS50 FTIR Spectrometer equipped with *in-situ* cells for transmission and DRIFTS modes. It is connected to an automated gas manfold to deliver the necessary gases to the reactor.

Gas Supply





Gas Patch Panel





Gas Flow Manifold



Facilities at SSRL to Date:

In-situ X-ray Spectroscopy/Diffraction Experiments

Co-ACCESS has a growing suite of *in-situ* catalysis cells available, and two Hiden mass spectrometers for effluent gas analysis. At the beamline we

have developed capability to safely and reliably deliver high pressure gases

to the *in-situ* cell. This involved an upgrade to the gas cabinet, a new high/ambient pressure manifold in the hutch, and a fully automated gas delivery system under LabView control. This is

schematically At the beamline we have illustrated in the developed capability to figure. We can safely and reliably deliver deliver H₂, CO and high pressure gases to He up to 80 bar for e.g. the in-situ cell.

> in-situ studies of Fischer-Tropsch catalysis from syngas.



In-situ Reactor



(mass spectrometer)





Enhanced Capabilities for XAS

Co-ACCESS is working together with the staff at SSRL to expand the current capabilities at the beamlines. Of particular recent interest is moving from step-scanning to continuous-scanning the photon energy on the XAS beamlines in collaboration with Oliver Müller. We have demonstrated that equivalent XAS data can be obtained in less than 10 minutes than conventionally takes over an hour to collect. This opens up many new exciting opportunities for time-resolved XAS at SSRL.

Outreach

We organized a workshop at the SSRL/LCLS Users Meeting in October, 2018 on: "Catalysis by Single Metal Atoms: What is All the Fuss About?"

We ran out first hands-on EXAFS analysis boot camp in December 2018. It was attended by graduate students from UCLA, UCSB and Univ. New Mexico.

> We invite any catalysis researcher to contact us prior to submitting a proposal to SSRL, or prior to their upcoming experiment. We can advise you at the appropriate level with the expressed aim of trying to maximize the success of your time at SSRL. We look forward to collaborating with you! simon.bare@slac.stanford.edu https://www-ssrl.slac.stanford.edu/content/science/chemistry-catalysis

CATALYSIS Laboratory



Co-ACCESS operates a 400 ft² wet chemistry laboratory specifically designed for catalysis studies. Typical activities in the lab include catalyst treatments, off-line testing of *in-situ/operando* cells for use at the beamlines, and prototyping of catalysis cells. The lab is equipped

- 6 ft and 4 ft fume hoods
- two gas cabinets for flammable and toxic
- an argon glove box
- ovens and furnaces.

The lab is fully stocked with a diverse array of the catalysis researcher and is available to collaborators.