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XPS above atmospheric pressure at POLARIS

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While many processes have been proposed to address climate change, from reducing the need for fossil fuels to changing the source of electricity, only one process can genuinely resolve the build-up of CO2 in the air: to synthesize fuel from the air. While a daunting task at the turn of the 20th century, the Haber-Bosch process accomplished a similar feat, converting nitrogen in the air to liquid ammonia for fertilizer. Today we require new tools to achieve more detailed knowledge of complex reactions on the relevant catalyst surfaces. To this end, POLARIS, the first high-pressure XPS, has been built. To measure the surfaces of catalysts at pressures over one atmosphere, we have made many technologic advances, discovering new and rediscovering old problems.

Since the installation of POLARIS at DESY in 2017, many experiments have taken place including, CO oxidation, methane oxidation, CO reduction, CO2 reduction, and N2 reduction. To reach the pressures required for these and other reactions, major shifts from conventional ambient pressure XPS are needed:
1. The aperture to the analyzer and the gap between the sample and aperture must be reduced to the micron scale to reduce scattering.
2. High energy x-rays must be used to reduce x-ray attenuation and provide enough kinetic energy for electrons to penetrate the gas.
3. Most importantly, the gas needs to flow from the analyzer to the chamber forming a virtual gas cell resulting in a much larger volume of gas required.

By incorporating these innovations, it is possible to measure XPS spectra at pressures over one bar. While the above process provides a framework, numerous other challenges arise, for instance, the lost surface sensitivity due to hard x-rays or the imprecise nature of maintaining micron-sized gaps between materials. Despite challenges, POLARIS has become a reliable tool for measuring the surface of catalysts in situ, providing the first look at the atomic species present during the most important catalytic reactions.

Ambient pressure XPS was first developed within years of the first XPS and had been limited to the millibar pressure range until POLARIS. At the core, POLARIS operates under the same process as all XPS; by using monochromatic x-rays of known energy to emit electrons, measure the kinetic energy and calculate the binding energy. Many adaptations needed to be made to build the high-pressure XPS, from determining the materials that can withstand the corrosive environment to designing a process to maintain a constant gap between the sample and aperture. Along with the development of the virtual gas cell, there are needed advances to the light source. Besides higher photon energies, highly focused light is required. The myriad problems and unique solutions have made POLARIS a complex yet highly exceptional instrument to measure XPS spectra approaching industrial pressures.

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