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Catalysis on glowing catalysts: Breaking the 1000oC limit in APXPS

Content

Whether heterogeneous, homogeneous, or enzymatic, the catalyst represents one of the pillars on which modern society is built. All over the planet, billions of times a second, at scales of thousands of tons, the catalyst-gas phase interface shapes the world we live in. The scientific community is actively pursuing to characterize this interface in its operating state, under relevant conditions by bridging the pressure and temperature gaps between the laboratory and real-world environments. However, the temperature gap has remained a challenge, due to technical constraints of size and performance limitations when transitioning from ultrahigh vacuum to mbar conditions. In this work, we present a recent advancement in the in-situ characterization techniques, in the form of a high-temperature (HT) heating stage that bridges the temperature gap in UHV, mbar, and atmospheric pressure conditions in ambient pressure x-ray photoelectron spectroscopy (APXPS). The proof-of-concept experiments involved the HT pretreatment of a Ni foil and its further use as a catalyst in hydrocarbon partial oxidation.

The study, performed at the ISS beamline, proved the feasibility of performing spectroscopic measurements at extremely high temperatures. It was enabled by the use of a newly developed heating stage capable of achieving and sustaining sample temperatures of over 1200oC under reaction conditions for extended periods of time (>96h of continuous use). The heating stage was complemented by an automated unit that allows precise PID-based temperature regulation and thermal effects monitoring. The experiments targeted the oxidation and reduction of a model polycrystalline nickel foil in mbar H2 and O2 environments, up to 1050oC. Some of the preliminary results on the temporal evolution of the Ni 3p and O1s spectra during the HT oxidation and reduction are shown in ***Figure 1***. We discovered significant oxygen-containing surface species (H2O, OH-, and defective NiO) in the photoemission spectra by following the usual pretreatment procedure in H2 <700oC. However, increasing the temperature to 1050oC completes the reduction process; with Ni3p and O1s spectral regions showing reference grade spectra for metallic Ni. The pretreatment method produced an ideal starting state for further catalytic studies in hydrocarbon partial oxidation. The spectroscopic results were complemented by an operando scanning electron microscopy (OSEM) study that makes use of a similar heating stage.

In summary, the results highlight the importance of the HT, in-situ-enabled heating stage with application in APXPS and OSEM. This work is intended to enable further surface science studies that would expand the knowledge on the catalyst-gas phase interface. Industrially relevant systems that require operation temperature in excess of 1000oC, such as ammonia and methane oxidation and coupling or the catalytic hydrocarbon combustion and reforming are envisioned to benefit from this development.

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