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Modulation excitation spectroscopy coupled with synchrotron X-ray methods to unravel dynamic processes on supported Pd catalysts

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Automotive catalysts operate under fast transients and periodic reaction conditions that are challenging to reproduce for mechanistic studies. Improved data evaluation is required to separate the contribution of dynamic species from that of typically overwhelming static signals. This is achieved by a demodulation technique that has been applied to synchrotron techniques in an effort to capture the structural changes under fast transient conditions. Examples are discussed for time-resolved ED-EXAFS of Pd/Al2O3 during CO-NO pulsing and time-resolved hard-XRD of Pd/CeZrO4 during CO-O2 pulsing. The demodulation algorithm generates a set of phase-resolved data where static envelopes and all other features responding at a different frequency than that of the external stimulation are removed. This processing also considerably reduces the noise. Hence, small features in the both the EXAFS spectra and XRD data can be then clearly resolved.
The whole approach provides a sort of surface sensitivity to both EXAFS and XRD. The phase-resovled EXAFS data permit to observe the formation of PdCx in a CO pulse. In the XRD experiment, obvious reflections of reduced Pd appear only in the phase-resolved data set, apparently exceeding the detection threshold of conventional XRD for particles in the nano-scale. The data allow recognizing that the observed subtle changes occur at different rates thus uncovering the detailed structural-dynamic behaviour of the system with unprecedented sensitivity.

Primary author: Dr FERRI, Davide (Empa)

Co-authors: Prof. WEIDENKAFF, Anke (Empa); Dr CHIARELLO, GIan Luca (Empa); Dr NEWTON, Mark (ESRF); Dr MATAM, Santhosh Kumar (Empa); Ms LU, Ye (Empa)

Presenters: Dr FERRI, Davide (Empa); Dr CHIARELLO, GIan Luca (Empa)

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