

3rd Workshop on the Simultaneous Combination of Spectroscopies with X-ray Absorption, Scattering and Diffraction Techniques



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A novel setup for combined in situ XAS, DRIFTS and MES

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In situ DRIFTS and XAS are two complementary techniques in catalysis, allowing a deeper insight into reaction mechanisms and the dynamic of redox processes. However, XAS provides only bulk information due to the penetration depth of X-rays. The combination with modulated excitation spectroscopy (MES) through periodic switch of gas composition, and phase sensitive detection (PSD), has demonstrated to be a powerful tool to bring surface sensitivity to XAS. MES exploits the PSD algorithm used to demodulate the time-resolved data to filter the signals of spectator species and the noise present in the time domain. This approach is here used to enhance the response of time-resolved XAS (and DRIFTS) to the subtle variation of the surface of metal oxides.

Existing examples of combined IR-XAS setups are adaptations of commercial DRIFT cells to X-rays, whose use is limited to transmission and to selected materials because of the long optical path length. Our new cell, consisting of a plug-flow design, offers a wide surface for IR collection and a variable thin sample thickness for transmission measurements of difficult highly absorbing materials. Moreover, MES is facilitated by the absence of dead volume around the sample contrary to commercial DRIFT cells. The IR beam will be shined over the samples by means of a proper set of mirrors and positioning motors, placed along the X-ray beam. Preliminary results will be presented for oxidation of volatile organic compounds on metal oxides.

Primary authors: Dr FERRI, Davide (Empa); Dr CHIARELLO, Gian Luca (Empa)

Co-authors: Prof. WEIDENKAFF, Anke (Empa); Mr ALTORFER, Heinz (Empa); Dr QUARONI, Luca (PSI); Dr NAACHTEGAL, Maarten (PSI)

Presenter: Dr FERRI, Davide (Empa)

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