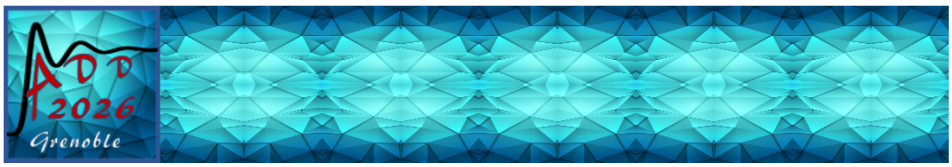

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Phase Sensitive Detection Analysis on Modulation Excitation PDF Data of Ni Based Catalysts

Content

Among catalysts employed for CO_2 methanation, Ni nanoparticles supported on γ-Al_2O_3 are the most widely used. Moreover, the transition from fossil to renewable energies means that an unstable supply of feedstock or energy must be considered when planning industrial processes and designing catalysts. Fluctuations in operating conditions can lead to structural changes and the formation of transient interfacial structures due to surface reconstruction processes. [1]
In-situ experiments involving catalytic cycling between catalysis and dropout conditions allow the power of modulation excitation (ME) experiments to be used. [2]
In this work, we present the first application of Phase Sensitive Detection (PSD) analysis to X-ray Pair Distribution Function (PDF) data, collected during in-situ catalysis experiments under ME conditions. [3]
To demonstrate the power of such ME-PDF analysis, we performed in-situ ME-PDF experiments on two Ni@Al_2O_3 catalysts (an industrial reference and an in-house synthetized one) under CO_2 methanation and full H_2 dropout conditions. In-situ synchrotron total scattering data were acquired at the I15-1 beamline at the Diamond Light Source in Didcot (Oxford, UK).
From the time-dependent PDF data, we were able to identify dynamic changes in the Ni lattice parameter and particle growth. The PSD transformed ME-PDF data revealed additional structural differences between the two catalysts. The PSD data showed that the synthetic route affects the structure of the catalysts. In the synthesised sample, an interaction between the metal and the support involving Al_2O_3 is observed. This interaction stabilises the Ni nanoparticles and prevents their oxidation. In contrast, the industrial reference catalyst showed no support involvement during the reaction, but exhibited a surface oxidation of the Ni nanoparticle during dropouts.
These structural observations were completely silent in the normal PDF analysis and became visible thanks to the application of the ME condition studied with PSD transformation.
In conclusion, ME-PDF analysis can have major impact in the field of catalysis in in-situ investigations at brilliant photon sources allowing ME experiments with fast cycling between operating conditions thanks to sub second measurement times of PXRD and PDF datasets in highly brilliant X-ray beams.

References:
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