Harnessing X-ray (Spectro)microscopy for Chemical Imaging of Heterogeneous Catalysts

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Heterogeneous catalysis is ubiquitous in chemical industry, with an estimated 90% of all chemical processes involving use of a catalyst at some stage [1]. These materials typically possess a complex hierarchical structure often spanning multiple length scales and containing various physical components, such as active metal nanoparticles (e.g. 1 to 10 nm), porous supports (nm-µm scale), macroscopic structure such as crystalline phases, pellets or monoliths (µm-mm scale). Furthermore, catalysts are functional materials, the performance of which is integrally related to their structure. Therefore to improve efficiency, sustainability and versatility for their numerous applications, it is optimal to develop an understanding of catalysts: (i) across multiple length scales covering all structural features of interest; (ii) in a spatially-resolved manner, avoiding the use of bulk or averaging analytical methods; (iii) under reaction conditions using ‘in situ’ or ‘operando’ analysis, allowing derivation of structure-activity relationships [2-3]. Structure-activity relationships in particular are a cornerstone of modern catalysis research, while the collection of spatially-resolved data avoids generalization and over-simplification of the structure observations made.

It is not surprising that the high flux, tunable energy and flexible experimental potential of synchrotron light sources play a crucial role in catalysis research, through the application of both X-ray spectroscopy and microscopy techniques. Modern hard X-ray microprobes and nanoprobes at third and upcoming fourth generation synchrotron light sources offer excellent opportunities for catalyst characterization [4], specifically through the development of so-called ‘chemical imaging’ methodologies - extracting chemically relevant information through the combination of highly focused X-rays with absorption, phase, fluorescence, diffraction or spectroscopic measurements, for example [5]. In many cases, the application of hard X-ray microscopy for chemical imaging is currently unexplored, despite the wealth of information which may be obtained.

This contribution will highlight several recent experiments in the field of exhaust gas catalysis, energy conversion, and nanostructured materials [6,7,8]. The specific focus will be on deriving chemically relevant data through hard X-ray microscopy on the µm and nm scale, such as metal oxidation state, crystalline structure, deactivation effects (e.g. sintering, structure collapse) and analysis of porosity. In most cases this data is acquired through tomographic imaging, meaning that the results are 3-dimensional renderings, offering numerous advantages in comparison to conventional bulk measurement. The design of reaction environments for in situ and operando chemical imaging in 2D and 3D will be demonstrated, along with case studies performed at the ESRF (Grenoble, France) and PSI (Villigen, Switzerland). A perspective on the imminent arrival of diffraction limited light sources and the implications for the field of X-ray microscopy in catalysis research will also be discussed.