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X-ray Physicochemical Imaging of Catalysts Under Working Conditions

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The imaging of catalysts and other functional materials under reaction conditions has advanced significantly in recent years. The combination of the computed tomography (CT) approach with methods such as X-ray diffraction (XRD), X-ray fluorescence (XRF), and X-ray absorption near edge spectroscopy (XANES) now enables local chemical and physical state information to be extracted from within the interiors of intact materials which are, by accident or design, commonly inhomogeneous. The spatially resolved signals obtained can reveal information that would otherwise be lost in bulk measurement. Such local signals are simpler to interpret since they are highly likely to contain fewer phases. Studying intact materials rather than idealised powders allows for behaviour under industrially relevant conditions to be observed. Furthermore the background signal from in situ apparatus / cell can be readily separated.

We show how such methods have been applied to understanding the behaviour of a number of catalytic systems at a range of length scales. Crucially we demonstrate that the obtained chemical and physical information can be correlated to catalytic activity and selectivity. At the small length scales, sample size and density allow for transmission of comparatively low energy signals allowing combination of XRF-CT and XANES-CT in conjunction with XRD-CT, enabling simultaneous multi-technique imaging. This combined approach has been used to characterise intact single catalytic particles (a 100 μm Mo promoted Pt/C catalyst under liquid phase hydrogenation operating conditions) and packed bed micro-reactors enabling identification of active species and correlation to performance. High energy XRD-CT enables large/dense objects to be studied. We show how this has been used to study the behaviour of industrial pellets (e.g. following the evolution of the cobalt phases in 3 mm diameter $\text{Co}/\text{Al}_2\text{O}_3$ catalyst under industrial conditions) and also to see inside working bulk reactors (6 mm catalytic membrane reactor device used for the oxidative coupling of methane; the reactor consists of a dense ceramic oxygen transport membrane containing a packed bed of $\text{Mn-Na-W}/\text{SiO}_2$). In the latter case the active state is captured and material is observed to form that is likely to impact on the long term performance of the reactor. Such information is vital to rational catalyst and reactor design that cannot be obtained by conventional bulk measurements.

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