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Revealing Catalyst Complexity in 3D with Synchrotron X-ray Nanotomography

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Catalysts are complex materials whose structure and reactive behaviour are closely interlinked. All length scales are relevant in catalysis, from metal nanoparticles, to porosity (micro- meso- and macropores), to global structure (e.g. grains, pellets or monoliths). While electron microscopy is well suited for analysis at (sub)nanometre resolution, this is often limited to small fields of view, thin sample lamella, and high vacuum conditions. Sample preparation is an important consideration, ideally delicate structural features should be preserved and measurements performed non-invasively. Developments in hard X-ray imaging using synchrotron radiation, particularly ptychographic X-ray computed tomography (PXCT), allow non-invasive measurement of large sample volumes with spatial resolutions unmatched by other X-ray microscopy methods. Here we demonstrate the derivation of structural information from two diverse catalyst systems using PXCT, (i) hierarchical nanoporous gold, (ii) Pt-alumina exhaust gas catalyst monoliths. In both cases micrometre-sized samples were measured, with 3D isotropic spatial resolutions of 13 and 35 nm, respectively. The 3D volumes obtained allowed direct and non-invasive quantification of catalyst surface area, pore size distribution and pore network topology. PXCT is highlighted as a method with uniquely high potential in catalysis research, which will furthermore benefit greatly from the advent of fourth generation synchrotron light sources.

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