

Dynamic in-situ ptychographic X-ray tomography with environmental control

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Dynamic material transformations such as crystallisation, sintering and phase separation underpin the performance and failure of functional materials, yet remain difficult to visualise in three dimensions under realistic operating conditions. We present an integrated platform for in-situ ptychographic X-ray tomography that combines X-ray ptychography, angularly sparse tomogram acquisition, dynamic 4D reconstruction and a custom environmental control and nano-positioning instrument. This setup enables the acquisition of quantitative electron-density tomograms of 100 µm wide samples with 10s of nanometre spatial and minute-scale temporal resolution, continuously across broad temperature ranges and gas atmospheres over extended periods. The approach is dose-efficient and compatible with complex sample geometries, allowing repeated imaging of the same volume throughout a transformation, and yields quantitative 4D datasets that are directly amenable to advanced analysis and multimodal workflows. We demonstrate these capabilities on two case studies. First, we follow the crystallisation of amorphous calcium carbonate from room temperature to 500°C, acquiring hundreds of tomograms that reveal discrete dehydration steps, heterogeneous nucleation pathways, and rare metastable phases, alongside defect-mediated Ostwald ripening in individual crystals. Second, we track the activation, mobility and deactivation of a supported catalyst under methane oxidation conditions from 25°C to 830°C, tracking the formation and evolution of over 100,000 nanoparticles within a hierarchically porous support, uncovering two concurrent catalyst deactivation modes.