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Sample design and preparation techniques for dynamic microstructural studies of high temperature electrochemical cells

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Understanding the dynamics of 3D microstructural change in high temperature electrochemical cells, primarily solid oxide fuel cells or electrolysers, is a pressing driving force for performing time resolved ex-situ, in-situ and in-operando nano-tomography and diffraction based experiments at synchrotron X-ray sources. These experiments must meet simultaneous challenging demands: precision beamline compatible samples that are stable at high temperature, supply of electric potential, and control of atmosphere. Correct sample design is an absolute necessity for experimental success. Here, the merits of possible sample configurations and environments are explored and evaluated against fabrication challenges and experimental feasibility. Experience with designing and performing experiments of selected configurations will be presented. Results of 3D nano-tomography of Ni-yttria stabilized zirconia (YSZ) fuel electrode microstructure evolution during Ni oxidation, reduction and annealing, and spatially resolved in-operando diffraction studies of YSZ electrolytes under at high polarization will be summarised.