MS40 Operando and in-situ crystallographic studies

MS40-1-2 3DED in situ single-crystal studies on submicron-sized particles during redox reactions in gas environments and electrochemical reactions #MS40-1-2

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## Abstract

Oxidation and reduction processes are important in the field of energy materials. Changes in crystal structures during such processes dictate ion conduction paths and reversibility, thus efficiency, capacity and lifetime of different technologies. Such structural changes are currently followed in situ using X-ray and neutron powder diffraction techniques, because the materials are usually only active as submicron particles, making in situ single crystal X-ray or neutron diffraction studies non-applicable. Although in situ powder diffraction can uncover very important structural changes, it can be hindered by peak overlap of different phases and peak broadening due to the small crystal sizes, leaving some structures unsolved or unrefined. Using instead electron diffraction allows performing in situ single crystal experiments on the individual particles within a powder sample, due to the much stronger interaction between electrons and matter, and allows uncovering information and determining structures out of reach of in situ powder diffraction techniques. In literature, in situ transmission electron microscopy is so far mainly used for imaging, but the possibilities of in situ electron diffraction and especially the advanced technique of 3DED (in which reciprocal space is reconstructed from a series of electron diffraction patterns taken at different angles such that the data can be used for solution and refinement, similar to other single crystal diffraction techniques) are unexplored. In this lecture we will demonstrate our pioneering results on in situ 3DED, where we have monitored how the crystal structures of materials change under oxidizing and reducing gas atmospheres, or under electrochemical oxidation and reduction.

## Oxidation of SrFeO2.5

