Following structure evolution of $SrFeO_x$ in redox reactions using *in situ* 3D electron diffraction

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Strontium iron oxide is a candidate for many different energy applications, including solid oxide fuel cells, chemical looping, and thermochemical energy storage. Upon the redox reactions, $SrFeO_x$ cycles between two end forms: an oxygen deficient form $SrFeO_{2.5}$ with a brownmillerite structure and an oxidized form SrFeO3-d with a perovskite structure. Two intermediate structures are reported from *ex situ* and *in situ* X-ray and neutron powder diffraction [1–3]. However, in real applications, submicron sized crystals are used and X-ray and neutron diffraction techniques are not able to access structural information on an individual submicron crystal. *In situ* 3D electron diffraction (3D ED) performed on a transmission electron microscope (TEM) is the only way to obtain single crystal data on all structural changes occurring during the actual redox reactions. Due to the single-tilt design of the environmental holders combined with the complexity of these structures, in-zone electron diffraction and high resolution imaging on random crystallites are unrealistic, but 3D ED does not require in zone orientation and could thus be successfully applied to gather structural data on the different phases.

We performed *in situ* oxidation of a brownmillerite $SrFeO_{2.5}$ crushed single crystal upon heating in an oxygen atmosphere in TEM using the sealed commercial holder. By acquiring 3D ED data at different steps of the reaction, we confirmed the formation of the perovskite $SrFeO_{3-\delta}$ structure, which we were able to reduce back to brownmillerite in a hydrogen atmosphere. The obtained data allowed us to derive the structures formed at different reaction steps, including the intermediate phases, resulting in new information about their crystal structures and microstructures. In my talk, I will compare the results of *in situ* 3D ED with the published data from X-ray and neutron diffraction, discuss the limitations of the method, and the next steps in improving *in situ* 3D ED in gas environments.

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