

# The Structural Study of $\text{La}_{0.9}\text{Sr}_{0.1}\text{Co}_{1-x}\text{Fe}_x\text{O}_{3-\Delta}$ Through in Situ Neutron and Synchrotron Diffraction

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To understand the structural evolution of the oxygen transport membranes  $\text{La}_{0.9}\text{Sr}_{0.1}\text{Co}_{1-x}\text{Fe}_x\text{O}_{3-\delta}$  ( $x = 0, 0.25, 0.75, 1$ ) (LSCFO) under industrially relevant conditions in situ neutron and synchrotron x-ray diffraction were collected and quantitatively studied via Rietveld refinements. As the temperature increased under reducing gas, the phase composition for all  $x$  values transitioned through intermediates phases, such as the Ruddlesden Popper phases  $\text{La}_2\text{CoO}_4$ ,  $\text{La}_2\text{FeO}_4$  and  $\text{La}_4\text{Co}_3\text{O}_{10}$ , ultimately reducing into a combination of metals or metal oxides ( $\text{La}_2\text{O}_3$ , Co, Fe, and SrO) at maximum temperature of 850 °C. Co rich LSCFO samples ( $x = 0, 0.25$ ) formed reduction products at earlier temperatures than Fe rich LSCFO ( $x = 0.75, 1$ ). Co rich LSCFO fully reduced into its reduction products; However, Fe rich LSCFO did not fully reduce during the set reduction period. The differing redox products provide mechanistic insights into what phases are available for catalysis at specific conditions. All systems showed redox cycling despite multiple structural phase being present throughout the redox process. Additionally, neutron and synchrotron x-ray diffraction data LSCFO compounds showed the formation of reduction products at different time and temperatures. It is hypothesized this difference is the result of differences in sample environments. This study gives insight both into the structural changes that accompany redox reactions in oxygen transport membranes as well as how to utilize in situ diffraction measurements to study these systems under extreme conditions.