NiO reduction studied by environmental transmission electron microscopy

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Resume : Nickel oxide reduction and subsequent Ni behavior under H2 atmosphere is of practical importance in the field of solid oxide fuel cells (SOFC) as it determines the structure of the anode electron conductor during operation. Despite extensive coverage in literature, some discrepancies remain, notably regarding NiO reduction kinetics and structural evolution. In situ reduction of an industrial NiO powder from JT Baker™ is performed under 1.3 mbar of H2 (2 mlN/min) in a differentially pumped FEI Titan 80-300 environmental transmission electron microscope (ETEM). Images, diffraction patterns and electron energy loss spectra (EELS) are acquired at different temperatures to monitor the structural and chemical evolution of the system. High-resolution ETEM is also performed during similar experiments. EELS analysis illustrates that reduction first proceeds quickly at temperatures below 400°C up to a reduced fraction of about 0.6, until the reaction is slowed down by water created upon reduction. Ni nucleation on NiO is observed to be either epitaxial in thin areas or randomly oriented in thicker regions. The growth of Ni crystallites creates pores within NiO grains to accommodate the volume shrinkage associated with reduction. Densification is then observed at temperatures higher than 550°C: pores created at lower temperatures disappear and Ni grains coarsen. This reorganization of Ni is detrimental to both the connectivity of the Ni catalyst and to the redox stability of the SOFC.