In situ TEM studies of redox cycling of a Ni-ScSZ cermet fuel cell anode

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In situ transmission electron microscopic (TEM) studies of a Ni(O) - Sc_2O_3-stabilized ZrO_2 (ScSZ; 10 mol% Sc_2O_3, 1 mol% CeO_2, 89 mol% ZrO_2) anode in a solid oxide fuel cell (SOFC) have been conducted at high temperatures under a hydrogen/oxygen gas atmosphere using an environmental transmission electron microscope (ETEM); the specimens were lifted out of the real SOFC by means of focused ion beam. When heating the NiO - ScSZ anode under a hydrogen atmosphere of 3 mbar in ETEM, nano-pores were formed at the grain boundaries and on the surface of NiO particles at around 400 °C due to the volume shrinkage accompanying the reduction of NiO to Ni. Moreover, densification of Ni occurred when increasing the temperature from 600 °C to 700 °C. High-magnification TEM images obtained in the early stages of NiO reduction revealed that the (111) planes of Ni grew almost parallel to the (111) planes of NiO. In the case of heating Ni - ScSZ under an oxygen atmosphere of 3 mbar in ETEM, oxidation of Ni starting from the surface of the particles occurred above 300 °C. All Ni particles became polycrystalline NiO after the temperature was increased to 800 °C. Volume expansion/contraction by mass transfer to the outside/inside of the Ni particles in the anode during repeated oxidation/reduction seems to result in the agglomeration of Ni catalysts during long-term SOFC operation. We emphasize that our in situ TEM observations will be applied to observe electrochemical reactions in SOFCs under applied electric fields.
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