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In situ reduction and oxidation of nickel from solid oxide fuel cells in a Titan ETEM

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Solid Oxide Fuel Cells (SOFC) common technology is based on anode-supported cells composed of nickel-yttria stabilized zirconia (Ni-YSZ) cermet. The nickel is in oxide state (NiO) during SOFC production and is reduced to metallic nickel during the first operation. The microstructure influences the SOFC electrochemical performance [1] as well as its stability for long-term use [2]. Oxidation of the nickel catalyst can occur at high fuel utilization and due to air leakage. The volume change from Ni to NiO can be detrimental for the thin supported electrolyte [3].

In situ transmission electron microscopy (TEM) enables to acquire further knowledge on the mechanisms behind the reduction and oxidation of nickel in the Ni-YSZ SOFC anode.

The *in situ* reduction and re-oxidation of the FIB prepared TEM lamellae is performed in a FEI Titan equipped with an environmental cell. Figure 1 presents a bright field micrograph of the anode microstructure before and during *in situ* reduction. The reduction started at 400°C under 1.4 mbar of hydrogen. The volume contraction due to NiO reduction is compensated by formation of nanoporosity in the Ni grain. Nanoporosity was previously observed by Waldbillig *et al.* during *ex situ* reduction of Ni-YSZ composite TEM lamellae [4].

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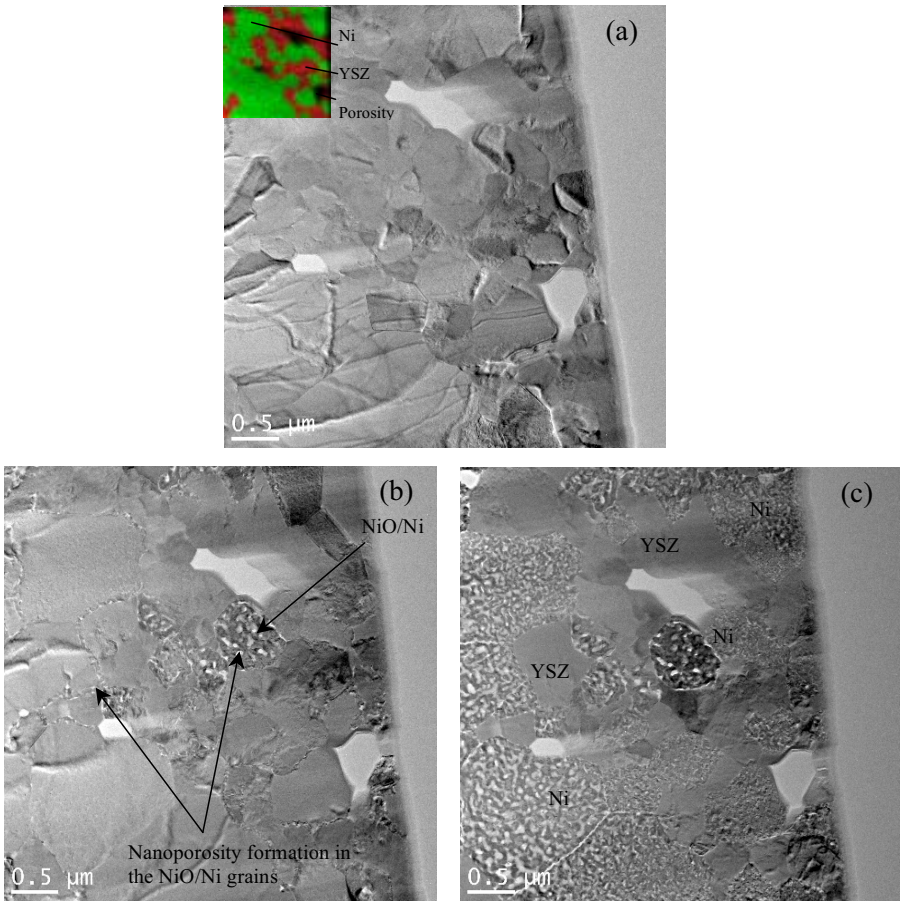

Figure 1. Bright field micrographs of the sample (a) at room temperature before reduction, (b) at 450°C under 1.4 mbar H_2 and (c) at 500°C under 1.4 mbar H_2. The EDX map of the micrograph is inserted in the image (a). YSZ grains are left unchanged by the reduction while the Ni grains exhibit internal nanoporosity (bright spots on the micrographs taken at 450°C and 500°C).