



## In Situ Reduction and Oxidation of Nickel from Solid Oxide Fuel Cells in a Titan ETEM

**Faes, Antonin; Jeangros, Quentin; Wagner, Jakob Birkedal; Hessler-Wyser, Aïcha; Van herle, Jane; Brisse, Annabelle; Dunin-Borkowski, Rafal E.**

*Published in:*  
Meeting Abstracts - Electrochemical Society

*Publication date:*  
2009

*Document Version*  
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

*Citation (APA):*  
Faes, A., Jeangros, Q., Wagner, J. B., Hessler-Wyser, A., Van herle, J., Brisse, A., & Dunin-Borkowski, R. E. (2009). In Situ Reduction and Oxidation of Nickel from Solid Oxide Fuel Cells in a Titan ETEM. In *Meeting Abstracts - Electrochemical Society* (pp. Abstract 1330). The Electrochemical Society.

---

### General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

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

A. Faes<sup>1,2</sup>, Q. Jeangros<sup>1</sup>, J.B. Wagner<sup>3</sup>, A. Hessler-Wyser<sup>1</sup>, J. Van herle<sup>2</sup>, A. Brisse<sup>5</sup>, R. Dunin-Borkowski<sup>3</sup>

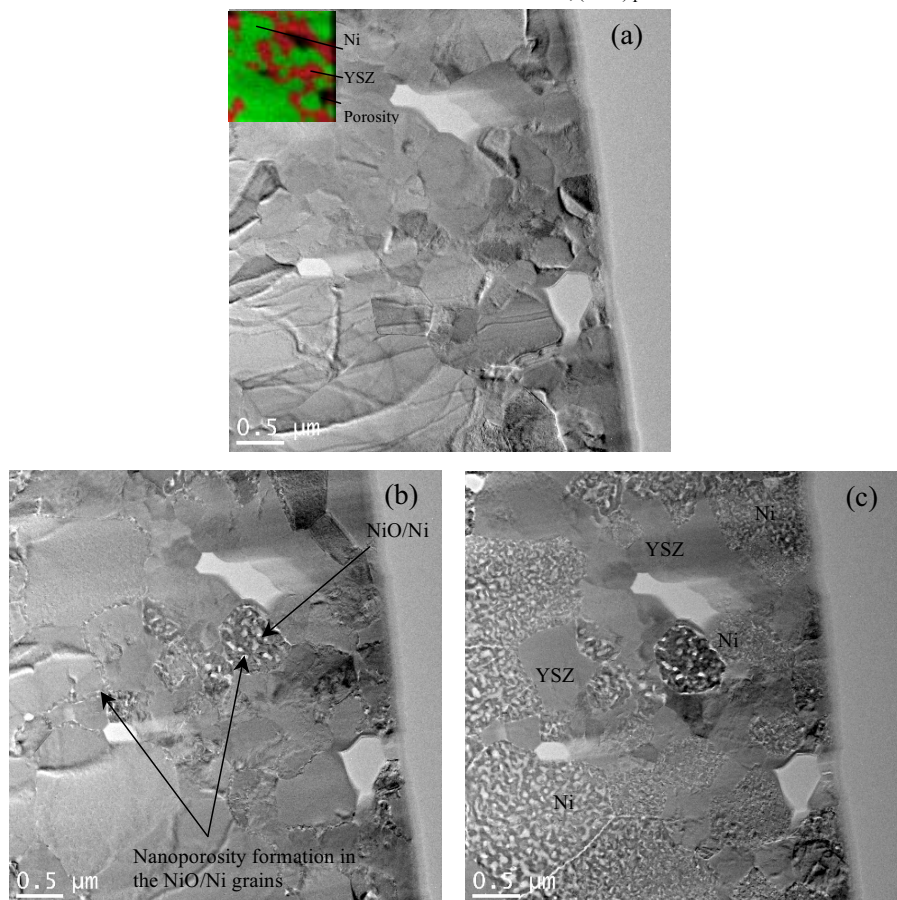
1. Interdisciplinary Centre for Electron Microscopy (CIME), Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015 Lausanne, Switzerland
2. Laboratory of Industrial Energy Systems (LENI), EPFL, CH-1015 Lausanne, Switzerland
3. Center for Electron Nanoscopy (CEN), Technical University of Denmark (DTU), DK-2800 Lyngby, Denmark
5. European Institute for Energy Research (EIFER), Emmy-Noether-Strasse 11, Karlsruhe, Germany

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].

1. S. C. Singhal, K. Kendall, High Temperature Solid Oxide Fuel Cell - Fundamentals, Design and Applications. (Elsevier, 2003).
2. D. Simwonis, F. Tietz, D. Stoeber, Solid State Ionics **132**, (2000) p241.
3. A. Faes, A. Nakajo, A. Hessler-Wyser, D. Dubois, S. Modena, A. Brisse, J. Van herle, J. Power Sources (2009) in press.
4. D. Waldbillig, A. Wood, D. G. Ivey, J. Power Sources **145**, (2005) p206.



**Figure 1.** Bright field micrographs of the sample (a) at room temperature before reduction, (b) at 450°C under 1.4 mbar H<sub>2</sub> and (c) at 500°C under 1.4 mbar H<sub>2</sub>. 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).