Research outputs:

**Complementary analyses of aging in a commercial LiFePO₄/graphite 26650 cell**

In this work we investigate the electrode degradation mechanisms in a commercial 2.5 Ah LiFePO₄/graphite 26650 cylindrical cell. Aged and fresh electrode samples were prepared by cycling two cells respectively five and 22 k times. Subsequently the cells were disassembled in a glovebox and the electrode samples were prepared for electrochemical testing in a 3-electrode setup, and for characterization with XRD, XPS and low-kV FIB/SEM tomography. A 1 μm thick CEI (cathode electrolyte interface) layer was observed at the electrode/electrolyte interface of the aged LiFePO₄ electrode. Relative to the fresh LiFePO₄ electrode, the aged electrode exhibited a larger series resistance which indicates the observed degradation layer increases the ionic resistance. In addition, micron-sized agglomerates, probably a mixture of carbonaceous material and decomposition products from the electrolyte, were observed at the electrode/electrolyte interface of the aged graphite electrode. These layers may contribute significantly to the loss of lithium inventory (LLI) in the cell, and to the loss of active material (LAM) in the graphite electrode. Low-voltage FIB/SEM tomography was used to detect local charging effects of graphite particles in the carbon electrode, an effect of poor dissipation of the electric charge to the ground after the sample interaction with the electron beam. The charging effects were primarily observed in the aged electrode and most of the locally charged particles were found to be close to the electrode/electrolyte interface, indicating a poorly percolating graphite network near this interface.

**General information**

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Original language: English
Keywords: Cathode electrolyte interface, Degradation mechanisms, Electrochemical impedance spectroscopy, Focused ion beam, Li-ion battery
Characterization of three dimensional transport networks in a long-term tested solid oxide electrolysis cell

Solid oxide electrolysis cells (SOEC) are a promising technology for energy conversion and storage. The SOEC cell consists of two electrodes on each side of a dense electrolyte. The electrodes are typically a two- or three-phase porous system, where the solid phases are responsible for conduction of electrons and oxygen ions and the pore phase allows transport of reactants and products to and from the electrochemically active sites. In this work we present a study of three dimensional transport networks in a long-term tested SOEC. The difference in the transport network quality at different cell locations was compared in terms of 3D microstructure parameters calculated from FIB serial sectioning image data. An advanced 3D transport network analysis was performed through simulations and geometrical calculations. Dramatic differences were observed between the gas inlet and outlet in the cell. The obtained 3D transport characteristics correlated well with the measured cell electrochemical performance.

Sample Design and Preparation Techniques for Dynamic Microstructural Studies of High Temperature Electrochemical Cells

Understanding the dynamics of 3D microstructural change in high temperature electrochemical cells, primarily solid oxide fuel cells or electrolyzers, is a pressing driving force for performing time resolved ex-situ, in-situ and in-operando nano-tomography and diffraction based experiments at synchrotron X-ray sources. These experiments must meet simultaneous challenging demands: precision beamline compatible samples that are stable at high temperature, supply of electric potential, and control of atmosphere. Correct sample design is an absolute necessity for experimental success. Here, the merits of possible sample configurations and environments are explored and evaluated against fabrication challenges and experimental feasibility. Experience with designing and performing experiments of selected configurations will be presented. Results of 3D nano-tomography of Ni-yttria stabilized zirconia (YSZ) fuel electrode microstructure evolution during Ni oxidation, reduction and annealing, and spatially resolved in-operando diffraction studies of YSZ electrolytes under at high polarization will be summarised.
Three dimensional characterization of nickel coarsening in solid oxide cells via ex-situ ptychographic nano-tomography

Nickel coarsening is considered a significant cause of solid oxide cell (SOC) performance degradation. Therefore, understanding the morphological changes in the nickel-yttria stabilized zirconia (Ni-YSZ) fuel electrode is crucial for the wide spread usage of SOC technology. This paper reports a study of the initial 3D microstructure evolution of a SOC analyzed in the pristine state and after 3 and 8 h of annealing at 850 °C, in dry hydrogen. The analysis of the evolution of the same location of the electrode shows a substantial change of the nickel and pore network during the first 3 h of treatment, while only negligible changes are observed after 8 h. The nickel coarsening results in loss of connectivity in the nickel network, reduced nickel specific surface area and decreased total triple phase boundary density. For the condition of this experiment, nickel coarsening is shown to be predominantly curvature driven, and changes in the electrode microstructure parameters are discussed in terms of local microstructural evolution.

General information
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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Paul Scherrer Institute
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BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 6.34 SJR 1.9 SNIP 1.667
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 6.3 SJR 1.964 SNIP 2.042
Web of Science (2014): Indexed yes
BFI (2013): BFI-level 1
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ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 5.04 SJR 2.282 SNIP 2.006
Web of Science (2012): Impact factor 4.675
ISI indexed (2012): ISI indexed yes
Web of Science (2012): Indexed yes
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 5.13 SJR 2.227 SNIP 2.172
Web of Science (2011): Impact factor 4.951
ISI indexed (2011): ISI indexed yes
A Decade of Solid Oxide Electrolysis Improvements at DTU Energy

Solid oxide electrolysis cells (SOECs) can efficiently convert electrical energy (e.g., surplus wind power) to energy stored in fuels such as hydrogen or other synthetic fuels. Performance and durability of the SOEC has increased orders of magnitude within the last decade. This paper presents a short review of the R&D work on SOEC single cells conducted at DTU Energy from 2005 to 2015. The SOEC improvements have involved increasing the of the oxygen electrode performance, elimination of impurities in the feed streams, optimization of processing routes, and fuel electrode structure optimization. All together, these improvements have led to a decrease in long-term degradation rate from \(\sim 40 \% / \text{kh}\) to \(\sim 0.4 \% / \text{kh}\) for steam electrolysis at -1 A/cm\(^2\), while the initial area specific resistance has been decreased from 0.44 \(\Omega\text{cm}^2\) to 0.15 \(\Omega\text{cm}^2\) at -0.5 A/cm\(^2\) and 750 °C.

General information

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Publication information
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A Physically-Based Equivalent Circuit Model for the Impedance of a LiFePO$_4$/Graphite 26650 Cylindrical Cell

In this work an Equivalent Circuit Model (ECM) is developed and used to model impedance spectra measured on a commercial 26650 LiFePO$_4$/Graphite cylindrical cell. The ECM is based on measurements and modeling of impedance spectra recorded separately on cathode (LiFePO$_4$) and anode (Graphite) samples, harvested from the commercial cell. Modeling of the single-electrode impedance spectra provided information about the electronic and ionic resistance in the porous composite electrodes, as well as the solid state diffusion. Focused Ion Beam (FIB)/Scanning Electron Microscopy (SEM) of anode and cathode samples was used to make 3-D maps of the electrode microstructures and to obtain microstructural data for the ECM. The complementary analysis was crucial for the resolution of the single electrode impedance parameters and the proposal and validation of a new equivalent circuit used to model the full commercial battery impedance.

General information
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Enhanced densification of thin tape cast Ceria-Gadolinium Oxide (CGO) layers by rheological optimization of slurries

Optimized CGO-based slurries are formulated and shaped into thin dense layers via a tape-casting process. The formulation is adjusted with respect to the rheological behaviour. The internal structure and flow properties of slurries are explored with the aim of identifying the required conditions to obtain thin dense CGO layers at reduced sintering temperatures (1200 °C). We demonstrate a correlation between the rheological properties of the slurries, the sintering behaviour and the microstructure of the resulting tapes. Remarkably, a dense CGO layer less than 20 μm thick is obtained with a non-congested slurry, having optimized ceramic loading and liquid-like behaviour.
Estimation of current constriction losses via 3D tomography reconstructions in electrochemical devices: a case study of a solid oxide cell electrode/electrolyte interface

In the present study, the methodology for accurate estimations of the current constriction resistance in solid state electrochemical devices via 3D tomography reconstructions is developed. The methodology is used to determine the current constriction resistances at the Ni:YSZ anode/YSZ electrolyte interface of a solid oxide fuel cell. The current constriction at this interface becomes increasingly important as thinner electrolyte layers are continuously being pursued.
for increased performance. Various possible scenarios have been illustrated on idealized geometries as a function of electrolyte thicknesses, from which it is clear, that for a given set of electrodes an optimal electrolyte thickness exist. Thus, increased performance by reduction of the electrolyte thickness is only feasible down to a certain thickness, after which, a lower performance is obtained on a further reduction of the electrolyte thickness. The obtained results on current constriction resistances from numerical calculations on a 3D reconstruction of a Ni:YSZ anode/YSZ electrolyte assembly is compared with existing models with analytical expressions. The comparison shows, that the assumptions of existing models are by far too simple and the models are therefore not applicable for technological relevant electrochemical devices.
Ex-situ tracking solid oxide cell electrode microstructural evolution in a redox cycle by high resolution ptychographic nanotomography

For solid oxide fuel and electrolysis cells, precise tracking of 3D microstructural change in the electrodes during operation is considered critical to understand the complex relationship between electrode microstructure and performance. Here, for the first time, we report a significant step towards this aim by visualizing a complete redox cycle in a solid oxide cell (SOC) electrode. The experiment demonstrates synchrotron-based ptychography as a method of imaging SOC electrodes, providing an unprecedented combination of 3D image quality and spatial resolution among non-destructive imaging techniques. Spatially registered 3D reconstructions of the same location in the electrode clearly show the evolution of the microstructure from the pristine state to the oxidized state and to the reduced state. A complete mechanical destruction of the zirconia backbone is observed via grain boundary fracture, the nickel and pore networks undergo major reorganization and the formation of internal voids is observed in the nickel-oxide particles after the oxidation. These observations are discussed in terms of reaction kinetics, electrode mechanical stress and the consequences of redox cycling on electrode performance.

General information
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Organisations: Department of Energy Conversion and Storage, Ceramic Engineering & Science, Imaging and Structural Analysis, Paul Scherrer Institute
Contributors: De Angelis, S., Jørgensen, P. S., Esposito, V., Hsiao Rho Tsai, E., Holler, M., Kreka, K., Abdellahi, E., Bowen, J. R.
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Scopus rating (2016): CiteScore 6.22 SJR 1.944 SNIP 1.5
Web of Science (2016): Indexed yes

BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 6.34 SJR 1.9 SNIP 1.667
Web of Science (2015): Indexed yes

BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 6.3 SJR 1.964 SNIP 2.042
Web of Science (2014): Indexed yes

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Scopus rating (2013): CiteScore 5.63 SJR 1.975 SNIP 2.137
Web of Science (2013): Impact factor 5.211
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes

BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 5.04 SJR 2.282 SNIP 2.006
Web of Science (2012): Impact factor 4.675
ISI indexed (2012): ISI indexed yes
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BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 5.13 SJR 2.227 SNIP 2.172
Web of Science (2011): Impact factor 4.951
ISI indexed (2011): ISI indexed yes
Web of Science (2011): Indexed yes

BFI (2010): BFI-level 1
Scopus rating (2010): SJR 2.294 SNIP 1.972
Web of Science (2010): Impact factor 4.29
Web of Science (2010): Indexed yes

BFI (2009): BFI-level 1
Scopus rating (2009): SJR 2.105 SNIP 1.785
Web of Science (2009): Indexed yes

BFI (2008): BFI-level 2
Scopus rating (2008): SJR 1.96 SNIP 1.713
Web of Science (2008): Indexed yes
Scopus rating (2007): SJR 1.587 SNIP 1.488
Web of Science (2007): Indexed yes
Scopus rating (2006): SJR 1.802 SNIP 2.223
Web of Science (2006): Indexed yes
Scopus rating (2005): SJR 1.656 SNIP 1.809
Web of Science (2005): Indexed yes
Scopus rating (2004): SJR 1.85 SNIP 1.805
Scopus rating (2003): SJR 1.66 SNIP 1.57
In operando studies of an yttria stabilized zirconia electrolyte supported symmetric solid oxide cell by Dark field X-ray Microscopy at ID06

Solid Oxide Cells are becoming a promising solution for sustainable and renewable power generation. Scandium doped Yttria Stabilized Zirconia is considered one of the best materials used as electrolyte because of its high ionic conductivity and great mechanical and chemical stability under operating conditions. Oxygen bubble formation at grain boundaries of ScYSZ near the anode/electrolyte interface has been observed as a degradation process when running in electrolysis mode at 800 - 900 °C for 24 - 72 hours at high current densities. X-ray diffraction can provide information about structural evolution at different depths of the cell during operation.

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Poster_Microsymposia_JXST_.pdf
Source-ID: 143955620
Research output: Research - peer-review › Journal article – Annual report year: 2017

In operando studies of ScYSZ electrolyte supported symmetric solid oxide cell by X-ray Diffraction at ESRF, ID06 Beamline

Solid Oxide Cells are becoming a promising solution for sustainable and renewable power generation. Scandium doped Yttria Stabilized Zirconia is considered one of the best materials used as electrolyte because of its high ionic conductivity and great mechanical and chemical stability under operating conditions. Oxygen bubble formation at grain boundaries of ScYSZ near the anode/electrolyte interface has been observed as a degradation process when running in electrolysis mode at 800 - 900 °C for 24 - 72 hours at high current densities. X-ray diffraction can provide information about structural evolution at different depths of the cell during operation.

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State: Published
Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Neutrons and X-rays for Materials Physics, Department of Physics, ESRF Beamline
The effects of long-term operation in electrolysis mode on the microstructure of Ni/YSZ electrodes were investigated. The electrode structures were investigated in "as reduced" state and after 9000 h of operation in a 25 cell stack. Microstructural data were obtained by scanning electron microscopy and focused-ion-beam serial sectioning. Microstructural characteristics were extracted by 1D and 3D methods. Significant microstructural changes were observed between the two cells analyzed. A significant loss of Ni in the active electrode is observed, from ~29% (by volume) in the reference cell to ~24% as well as a coarsening of the Ni particle sizes. The long-term tested cell shows lower percolating triple phase boundary density (0.76 µm/µm³) than the un-tested reference (2.0 µm/µm³). This reduction is mainly due to the loss of triple phase boundary percolation through the Ni phase where a reduction from a percolation degree above 90% to ~50% is observed.
Microstructural Characterization of Ni/YSZ Electrodes In a Solid Oxide Electrolysis Stack Tested for 9000 Hours

General information
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Organisations: Department of Energy Conversion and Storage, Mixed Conductors, Imaging and Structural Analysis, Applied Electrochemistry
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Publication date: 2017

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Using Dark Field X-Ray Microscopy To Study In-Operando Yttria Stabilized Zirconia Electrolyte Supported Solid Oxide Cell

Dark Field X-Ray Microscopy is a promising technique to study the structure of materials in nanometer length scale. In combination with x-ray diffraction technique, the microstructure evolution of Yttria Stabilized Zirconia electrolyte based solid oxide cell was studied running at extreme operating conditions.

General information
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A Decade of Improvements for Solid Oxide Electrolysis Cells. Long-Term Degradation Rate from 40%/Kh to 0.4 % Kh

Solid oxide electrolysis cells (SOEC) have the potential for efficient large-scale conversion from electrical energy to chemical energy stored in fuels, such as hydrogen or synthetic hydrocarbon fuels by use of well-known catalysis processes. Key issues for the break-through of this technology are to provide inexpensive, reliable, high performing and long-term stable SOEC for stack and system applications. At DTU Energy (formerly Department of Fuel Cells and Solid State Chemistry, Risø National Laboratory), research within SOEC for more than a decade has led to long-term degradation rates on cell level being improved from 40 %/kh to 0.4 %/kh for tests at -1 A/cm² (figure 1). In this paper, we review the key findings and highlight different performance and durability limiting factors that have been discovered, analyzed and addressed over the years to reach the tremendous increase in long-term stability for SOEC as illustrated by
the cell tests in figure 1.

A TEM study of morphological and structural degradation phenomena in LiFePO₄-CB cathodes: Morphological and structural degradation in LiFePO₄-CB cathodes

LiFePO₄-based cathodes suffer from various degradation mechanisms, which influences the battery performance. In this paper, morphological and structural degradation phenomena in laboratory cathodes made of LiFePO₄ mixed with carbon black (CB) in a 1 mol/L LiPF₆ in EC:DMC (1:1 by weight) electrolyte are investigated by transmission electron microscopy at various preparation, assembling, storage, and cycling stages. High-resolution transmission electron microscopy imaging shows that continuous SEI layers are formed on the LiFePO₄ particles and that both storage and cycling affect the formation. Additionally, loss of CB crystallinity, CB aggregation, and agglomeration is observed. Charge–discharge curves and impedance spectra measured during cycling confirm that these degradation mechanisms reduce the cathode conductivity and capacity.

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Current Constriction at Electrode/Electrolyte Interfaces in Solid Oxide Cell Electrochemical Devices Calculated Via 3D Reconstructions

Electrochemical devices such as batteries, fuel cells, electrolyzers, electrochemical reactors and electrochemical sensors are important technologies for the present and the future society. For further improvement or maturing of the various technologies it is important to understand, characterize and minimize the different losses within the devices.

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Electrodeposition of metallic 3D surface-profiles for superconductor tapes

Electrodeposition of metallic 3D surface-profiles for superconductor tapes

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Contributors: Wulff, A. C., Jørgensen, P. S., Nielsen, P. H., Hansen, J. O. B.
Number of pages: 1
Publication date: 2016
Peer-reviewed: Yes
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http://www.sustain.dtu.dk/

Electron microscopy investigations of changes in morphology and conductivity of LiFePO4/C electrodes

In this work we study the structural degradation of a laboratory Li-ion battery LiFePO4/Carbon Black (LFP/CB) cathode by various electron microscopy techniques including low kV Focused Ion Beam (FIB)/Scanning Electron Microscopy (SEM) 3D tomography. Several changes are observed in FIB/SEM images of fresh and degraded cathodes, including cracks in the LFP particles, secondary disconnected particles, and agglomeration of CB. Low voltage (1 kV) SEM images show that the CB agglomerates have a different brightness than the fresh CB, due to charging effects. This suggests that the electronic conductivity of the CB agglomerates is low compared to that of the fresh CB particles. HRTEM analysis shows that fresh CB particles are quasi crystalline, whereas the LFP/CB interface in the degraded electrode shows amorphous carbon surrounding the LFP particles. The presence of the amorphous carbon is known to impede the electronic conductivity and thereby decreasing percolation in the cathode and reducing the electrode capacity.

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Web of Science (2017): Indexed yes
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Scopus rating (2016): CiteScore 6.22 SJR 1.944 SNIP 1.5
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 6.34 SJR 1.9 SNIP 1.667
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 6.3 SJR 1.964 SNIP 2.042
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Web of Science (2013): Impact factor 5.211
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Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 5.04 SJR 2.282 SNIP 2.006
Web of Science (2012): Impact factor 4.675
ISI indexed (2012): ISI indexed yes
Web of Science (2012): Indexed yes
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Scopus rating (2011): CiteScore 5.13 SJR 2.227 SNIP 2.172
Web of Science (2011): Impact factor 4.951
ISI indexed (2011): ISI indexed yes
Web of Science (2011): Indexed yes
BFI (2010): BFI-level 1
Scopus rating (2010): SJR 2.294 SNIP 1.972
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Web of Science (2010): Indexed yes
BFI (2009): BFI-level 1
Scopus rating (2009): SJR 2.105 SNIP 1.785
Web of Science (2009): Indexed yes
BFI (2008): BFI-level 2
Scopus rating (2008): SJR 1.96 SNIP 1.713
Web of Science (2008): Indexed yes
Scopus rating (2007): SJR 1.587 SNIP 1.488
Web of Science (2007): Indexed yes
Scopus rating (2006): SJR 1.802 SNIP 2.223
Web of Science (2006): Indexed yes
Scopus rating (2005): SJR 1.656 SNIP 1.809
Web of Science (2005): Indexed yes
Scopus rating (2004): SJR 1.85 SNIP 1.805
Scopus rating (2003): SJR 1.66 SNIP 1.57
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Web of Science (2002): Indexed yes
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Electronic versions: Electron_microscopy_investigations.pdf
Improved electrodes and gas impurity investigations on alkaline electrolysers

Alkaline water electrolysis for hydrogen production is a well-established technique, but some technological issues regarding the coupling of alkaline water electrolysis and Renewable Energy Sources (RES) remain to be improved.

Relaxation of stresses during reduction of anode supported SOFCs

To assess the reliability of solid oxide fuel cell (SOFC) stacks during operation, the stress field in the stack must be known. During operation the stress field will depend on time as creep processes relax stresses. This work reports further details on a newly discovered creep phenomenon, accelerated creep, taking place during the reduction of a Ni-YSZ anode. This relaxes stresses at a much higher rate (~×104) than creep during operation. Thus, the phenomenon of accelerated creep during reduction has to be considered both in the production of stacks and in the analysis of the stress field in a stack based on anode supported SOFCs. Accelerated creep has previously been studied in experiments with simultaneous loading and reduction. The hypothesis for the phenomenon centers around a significant softening of the Ni phase, which amongst other should lead to a significant relaxation of internal stresses in the Ni(O)-YSZ microstructure. The internal residual stresses can be anticipated due to the different thermal contractions of the two phases from the sintering temperature to the reduction temperature. It was thus concluded that with the recorded high creep rates, the stresses in a cell at the time of reduction should decrease significantly over minutes. In this work these internal stresses are measured in-situ before and after the reduction by use of X-ray diffraction. This is done by determining the elastic micro-strains (correlating to the stresses), which are assessed from the widening of the Bragg peaks. This enables us to determine the stresses in the different phases locally inside the microstructure of the composite Ni(O)-YSZ anode. Furthermore, the residual stresses have been modeled during cool-down from the reduction temperature. The stresses have been assessed by use of a combination of a 3D microstructural reconstruction by FIB-SEM, a microstructural finite element model and analytical homogenization considerations. A significant decrease of stresses is observed through the reduction as predicted, which partly confirms the hypothesis for the accelerated creep. Also, a significant relaxation of stresses to lower temperatures (~300°C) was also found. This was confirmed by the models, but is however not consistent with previous recorded coefficients of thermal expansion.
Thermo-Chemo-Mechanical Response of Solid Oxide Cells during Reduction and Cooling

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Contributors: Chatzichristodoulou, C., Charlas, B., Kwok, K., Jørgensen, P. S., Norby, P., Hendriksen, P. V., Frandsen, H. L.
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Colloidal stabilization of cerium-gadolinium oxide (CGO) suspensions via rheology
A rheological method based on the analysis of the flow index is proposed for the optimization of ceramic suspensions with respect to dispersant-ceramic affinity, dispersant concentration, and ceramic loading. The single-flow index (SFI) feature was identified as the criterion defining the optimized colloidal stable state. The method was applied to explore the ability of four commercial dispersants (acidic affine, neutral, basic affine, and polyvinylpyrrolidone (PVP)) to disperse cerium-gadolinium oxide (CGO) in ethanol. Only the acidic affine and the PVP dispersants were found to efficiently disperse the CGO powder. The acidic affine dispersant was further demonstrated to impart superior packing properties due to the formation of a thinner monolayer around the ceramic surface. CGO suspensions using the acidic affine at optimized amount were prepared and processed via tape casting. The resulting green tapes exhibited uniform and high packing density, producing a theoretical density in the sintered tapes of ca. 97-98%.

General information
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Organisations: Department of Energy Conversion and Storage, Ceramic Engineering & Science, Imaging and Structural Analysis, Mixed Conductors
Contributors: Marani, D., Sudireddy, B. R., Bentzen, J. J., Jørgensen, P. S., Kiebach, R.
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Scopus rating (2017): CiteScore 3.55 SJR 1.068 SNIP 1.698
Web of Science (2017): Impact factor 3.794
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BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 3.25 SJR 1.142 SNIP 1.888
Web of Science (2016): Impact factor 3.454
Web of Science (2016): Indexed yes
Computation of Effective Steady-State Creep of Porous Ni–YSZ Composites with Reconstructed Microstructures

This paper investigates the effective steady-state creep response of porous Ni–YSZ composites used in solid oxide fuel cell applications by numerical homogenization based on three-dimensional microstructural reconstructions and steady state creep properties of the constituent phases. The Ni phase is found to carry insignificant stress in the composite and has a negligible role in the effective creep behavior. Thus, when determining effective creep, porous Ni–YSZ composites can be regarded as porous YSZ in which the Ni phase is counted as additional porosity. The stress exponents of porous YSZ are the same as that of dense YSZ, but the effective creep rate increases by a factor of 8–10 due to porosity. The relationship of creep rate and volume fraction of YSZ computed by numerical homogenization is underestimated by most existing analytical models. The Ramakrishnan–Arunchalam creep model provides the closest approximation among all analytical models.

General information
State: Published
Organisations: Department of Energy Conversion and Storage, Mixed Conductors, Imaging and Structural Analysis
Contributors: Kwok, K., Jørgensen, P. S., Frandsen, H. L.
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BFI (2018): BFI-level 1
Web of Science (2018): Indexed yes
BFI (2017): BFI-level 1
Scopus rating (2017): CiteScore 3.06 SJR 0.95 SNIP 1.325
Web of Science (2017): Impact factor 2.956
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 2.77 SJR 1.028 SNIP 1.428
Web of Science (2016): Impact factor 2.841
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 2.71 SJR 0.995 SNIP 1.37
Web of Science (2015): Impact factor 2.787
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 2.78 SJR 1.167 SNIP 1.595
Web of Science (2014): Impact factor 2.61
Web of Science (2014): Indexed yes
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 2.52 SJR 1.16 SNIP 1.48
Web of Science (2013): Impact factor 2.428
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 2.39 SJR 1.271 SNIP 1.5
Web of Science (2012): Impact factor 2.107
ISI indexed (2012): ISI indexed yes
Web of Science (2012): Indexed yes
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 2.45 SJR 0.958 SNIP 1.447
Web of Science (2011): Impact factor 2.272
ISI indexed (2011): ISI indexed yes
Degradation Studies on LiFePO$_4$ cathode

In this paper we examine a laboratory LiFePO$_4$ (LFP) cathode and propose a simple model that predicts the electrode capacity as function of C-rate, number of cycles and calendar time. Microcracks were found in Li$_{1-x}$FePO$_4$ particles in a degraded LFP electrode and low-acceleration voltage (1 kV) FIB/SEM analysis allowed us to obtain phase contrast between FePO$_4$ and LiFePO$_4$. The evolution of micro-cracks is expected to increase the concentration of LFP particles which are not electronically accessible and thus cause a loss in capacity.

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Organisations: Department of Energy Conversion and Storage, Applied Electrochemistry, Imaging and Structural Analysis, Atomic scale modelling and materials, Department of Electrical Engineering
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BFI (2017): BFI-level 1
Scopus rating (2017): CiteScore 0.44 SJR 0.225 SNIP 0.252
Dictionary Based Segmentation in Volumes

We present a method for supervised volumetric segmentation based on a dictionary of small cubes composed of pairs of intensity and label cubes. Intensity cubes are small image volumes where each voxel contains an image intensity. Label cubes are volumes with voxelwise probabilities for a given label. The segmentation process is done by matching a cube from the volume, of the same size as the dictionary intensity cubes, to the most similar intensity dictionary cube, and from the associated label cube we get voxel-wise label probabilities. Probabilities from overlapping cubes are averaged and hereby we obtain a robust label probability encoding. The dictionary is computed from labeled volumetric image data based on weighted clustering. We experimentally demonstrate our method using two data sets from material science—a phantom data set of a solid oxide fuel cell simulation for detecting three phases and their interfaces, and a tomogram of a glass fiber composite used in wind turbine blades for detecting individual glass fibers.

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Organisations: Department of Applied Mathematics and Computer Science, Image Analysis & Computer Graphics, Department of Wind Energy, Composites and Materials Mechanics, Department of Energy Conversion and Storage, Imaging and Structural Analysis
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Dictionary Based Segmentation in Volumes
Method for supervised segmentation of volumetric data. The method is trained from manual annotations, and these annotations make the method very flexible, which we demonstrate in our experiments. Our method infers label information locally by matching the pattern in a neighborhood around a voxel to a dictionary, and hereby accounts for the volume texture.

General information
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Effect of Aging on the Electrochemical Performance of LSM-YSZ Cathodes
Investigations of degradation mechanisms of solid oxide fuel cells are crucial for achieving a widespread commercialization of the technology. In this work, electrochemical impedance spectroscopy (EIS) was applied for studying the aging effect on LSM-YSZ cathodes exposed to humidified air at 900°C for up to 3000 h. EIS spectra were fitted by a transmission line model for estimating relevant parameters associated with the LSM/YSZ charge transfer reaction and the oxide ion conduction through the YSZ network. For the reference non-aged sample, the ionic conductivity values are the expected ones for YSZ with 1 eV activation energy and no dependency on oxygen partial pressure (pO2), while the charge transfer resistance presents an activation energy of 1.6 eV and is proportional to (pO2)−0.31±0.08. These values agree with those reported in literature, validating the used model. The charge transfer resistance shows no clear tendency with aging time, while the ionic conductivity decreases up to ~79%. Accordingly, the electrochemically active thickness contracts from 60–135 μm to 45–60 μm. The changes observed in the cathode transport and electrochemical properties are mostly explained by the evolution of the phases present in agreement with results previously reported in the literature.

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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Consejo Nacional de Investigaciones Científicas y Técnicas, Meneta Advanced Shims Technology A/S
Contributors: Baqué, L. C., Jørgensen, P. S., Zhang, W., Hansen, K. V., Segaard, M.
Pages: F971–F981
Publication date: 2015
Peer-reviewed: Yes
Enabling Flexible Polymer Tandem Solar Cells by 3D Ptychographic Imaging

The realization of a complete tandem polymer solar cell under ambient conditions using only printing and coating methods on a flexible substrate results in a fully scalable process but also requires accurate control during layer formation to succeed. The serial process where the layers are added one after the other by wet processing leaves plenty of room for error and the process development calls for an analytical technique that enables 3D reconstruction of the layer stack with the possibility to probe thickness, density, and chemistry of the individual layers in the stack. The use of ptychography on a complete 12-layer solar cell stack is presented and it is shown that this technique provides the necessary insight to enable efficient development of inks and processes for the most critical layers in the tandem stack such as the recombination layer where solvent penetration in fully solution processed 12-layer stacks is critical in eleven of the steps.

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Organisations: Department of Energy Conversion and Storage, Functional organic materials, Imaging and Structural Analysis, Deutsches Elektronen-Synchrotron
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Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 12.96 SJR 6.515 SNIP 2.14
Web of Science (2016): Impact factor 16.721
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 14.2 SJR 6.219 SNIP 2.546
Web of Science (2015): Impact factor 15.23
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 15.27 SJR 6.668 SNIP 2.942
Li-ion batteries find widespread use in many electricity storage applications, from portable devices to electric vehicles [1-3], and LiFePO4 (LFP) is one of the most common cathodes because of its long durability and high safety [4,5] but, since its low ionic and electronic conduction, it is always mixed with carbon black (CB) additives to increase electronic percolation in the electrode. Focused Ion Beam (FIB)/Scanning Electron Microscopy (SEM) Tomography is one of the most used techniques for the study of the three-dimensional microstructure of porous electrodes [6-8]. Imaging at low-kV has been shown to be an excellent technique for studying electron percolation in Ni-network in solid oxide fuel cells [9]. In this work we study the degradation process that occurs in a LFP/C electrode by Low-kV FIB/SEM Tomography, using the low-voltage percolation technique to identify compositional changes in the CB network in three-dimensions. FIB/SEM images of a fresh and degraded cathode are compared and LFP grains are seen to crack with cycling, resulting in the formation of secondary disconnected particles with increased ionic resistivity; CB particles are instead observed to agglomerate, reducing the electrochemically active surface area. Using low voltage imaging (1 kV) a significant fraction of the large carbon agglomerates found in the aged electrode show a higher secondary electron yield compared to the fresh CB particles at low accelerating voltage. This suggests that degradation occurs both due to morphological changes and due to amorphous-crystalline phase transitions in the carbon network, resulting in non-percolating CB agglomerates.

Figure 1 shows lateral Everhart – Thornley (E-T) and in-lens detector images of fresh (a, b) and degraded cathode (c, d) collected after FIB slicing. White grains are LFP, black particles are CB additive and grey regions are pores infiltrated with silicon resin to improve phase contrast [7]. The CB network appears entirely dark in the in-lens detector image of the fresh electrode (Fig. 1b). It is possible to notice some charging effects from the insulating silicon resin. The in-lens detector image of the degraded electrode (Fig. 1d) is instead characterized by the presence of big carbon agglomerates (red rings) which are brighter because they charge as the electron beam hit them. This indicates a lower electric conductivity. 3D reconstruction of the entire network (Fig. 2) revealed that the carbon phase with lower electric conductivity accounted for approximately 25% of the volume of the total carbon in the sample. This of course affects electrode capacity since a reduced electron percolation in the CB network impedes (de)lithiation process of LFP particles. References - M. Armand, J. M. Tarascon, Nature, 451, 652-657 (2008). - B. Scrosati, J. Hassoun, and Y.K. Sun, Energy Environ. Sci., 4, 3287-3295 (2011). - J.M. Tarascon, M. Armand, Nature, 414, 359-367 (2001). - A.K. Padhi, K.S. Nanjundaswamy, and J. B. Goodenough, J. Electrochem. Soc., 144(4), 1188-1194 (1997). - Y. Wang, P. He, and H. Zhou, Energy Environ. Sci., 4, 805-817 (2010). - T. Hutzenlaub et al., Electrochemical and Solid-State Letters, 15 (3), A33-A36 (2012). - M. Ender et al, J. Electrochem. Soc., 159(7), A972-A980 (2012). - Z. Liu et al., J. Power Sources, 227, 267-274 (2013). - K. Thydén, Y.L. Liu, and J.B. Bilde-Sørensen, Solid State Ionics, 178, 1984-1989 (2008). [Figure]
Low-voltage FIB/SEM Tomography for 3D Microstructure Evolution of LiFePO₄/C Electrode

This work presents an investigation of the degradation mechanisms that occur in LiFePO₄/C battery electrodes during charge/discharge cycling. Impedance spectra were measured on a fresh electrode and an electrode aged by cycling. The spectra were modeled with an equivalent circuit which indicates that both the ionic and electronic pathways in the electrode were negatively affected by the cycling. Focused Ion Beam/Scanning Electron Microscopy (FIB/SEM) tomography of both electrodes shows that cycling causes agglomerations of Carbon black (CB). In addition to this, Low-voltage FIB/SEM revealed non-conductive CB in the aged electrode.

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Scopus rating (2017): CiteScore 0.44 SJR 0.225 SNIP 0.252
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 0.4 SJR 0.228 SNIP 0.253
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 0.36 SJR 0.211 SNIP 0.244
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 0.36 SJR 0.212 SNIP 0.234
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 0.27 SJR 0.192 SNIP 0.231
ISI indexed (2013): ISI indexed no
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 0.29 SJR 0.241 SNIP 0.26
ISI indexed (2012): ISI indexed no
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 0.36 SJR 0.261 SNIP 0.28
ISI indexed (2011): ISI indexed no
BFI (2010): BFI-level 1
Scopus rating (2010): SJR 0.249 SNIP 0.251
BFI (2009): BFI-level 1
Scopus rating (2009): SJR 0.242 SNIP 0.27
BFI (2008): BFI-level 1
Scopus rating (2008): SJR 0.254 SNIP 0.255
Scopus rating (2007): SJR 0.213 SNIP 0.206
Ni-hydroxide growth in vacuum plasma sprayed electrodes for alkaline electrolysis

General information
State: Published
Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Jilin University, Deutsches Zentrum Für Luft- und Raumfahrt
Contributors: Bentzen, J. J., Zhang, W., Jørgensen, P. S., Bowen, J. R., Reissner, R.
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Ni_hydroxide_growth_abstract.pdf
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RES Hydrogen: efficient pressurised alkaline electrolyser

The RESelyser project addresses issues associated with coupling alkaline electrolysis to renewable energy sources such as electrode stability and gas purity by implementing improved electrodes and a new separator membrane concept. The project aims to improve performance, operation pressure and reduce system cost. The project supports DTU Energy's activities on electrodes within the larger FCH-JU project. The overall project demonstrated: improved electrode efficiency also during cyclic operation, safe gas purity at a system pressure of 30 bar, 10 kW stack operation and estimated system costs including BoP. Investigation of cathodes revealed highly heterogeneous microstructures and 3D microstructure quantification methods were developed. Nanometre scale -Ni(OH)2 formation was identified on tested cathode surfaces and is considered a potential degradation mechanism that is not presently well understood.

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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis
Contributors: Bowen, J. R., Bentzen, J. J., Jørgensen, P. S., Zhang, W.
Number of pages: 48
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Triple phase boundary specific pathway analysis for quantitative characterization of solid oxide cell electrode microstructure

The density and percolation of Triple phase boundary sites are important quantities in analyzing microstructures of solid oxide fuel cell electrodes from tomography data. However, these measures do not provide descriptions of the quality of the TPB sites in terms of the length and radius of the pathways through which they can be reached. New methods for performing TPB specific pathway analysis on 3D image data are introduced, analyzing the pathway properties of each TPB site in the electrode structure. The methods seek to provide additional information beyond whether the TPB sites are percolating or not by also analyzing the pathway length to the TPB sites and the bottleneck radius of the pathway. We show how these methods can be utilized in quantifying and relating the TPB specific results to cell test data of an electrode reduction protocol study for Ni/Scandia-and-Yttria-doped-Zirconia (Ni/ScYSZ) anodes. A study of the TPB density and particle size distribution alone did not provide an explanation for the differences observed in electrode performance. However, the analysis of pathway lengths to the TPBs and the bottleneck radii to reach these TPB sites provided valuable microstructural insight that supported the findings from the electrochemical characterization of the Ni/ScYSZ anodes.
Assessment of full ceramic solid oxide fuel cells based on modified strontium titanates

Today’s solid oxide fuel cells based on composite Ni-cermet anodes have been developed up to reasonable levels of performance and durability. However, especially for small combined heat and power supply systems, known failure mechanisms e.g. re-oxidation, sulfur tolerance and coking have stimulated the development for full ceramic anodes based on strontium titanates. Furthermore, the Ni-cermet is primarily a hydrogen oxidation electrode and efficiency losses might occur when operating on carbon containing fuels.

In the European project SCOTAS-SOFC full ceramic cells comprising CGO/Ni infiltrated SrTiO3 anodes, and LSM/YSZ...
Cathodes have been developed and tested as single 5 x 5 cm² cells and up to 100 cm² circular cells. The initial performance exceeded 0.4 W/cm² at 850 °C and redox tolerance has been proven. The cell concept provides flexibility with respect to the used electro-catalysts and various infiltrated metals including Ni and Ru have been studied. Stable power output has been observed for Ru and Ni-CGO as infiltrate. While redox tolerance is maintained, both types of cells degrade rapidly under exposure to sulfur. An initial assembly of a 60 cell stack in a one kW Hexis Galileo system indicates the necessity for further stack design adaptation in order to account for the lower electronic conductivity compared to Ni-cermet based cells.

Degradation Studies on LiFePO₄ cathode
Lithium-ion batteries are a promising technology for automotive application, but limited performance and lifetime is still a big issue. The aim of this work is to study and address degradation processes which affect LiFePO₄ (LFP) cathodes - one of the most common cathodes in commercial Li-ion batteries. In order to evaluate how the LFP cathode is affected by C-rate a LFP working electrode, Lithium metal foil counter electrode and Lithium metal reference electrode was tested in a 3-electrode setup with a standard 1M LiPF₆ in 1:1 EC/DMC electrolyte and glass fiber separator. The working electrode/counter electrode was subjected to several charge/discharge cycles between 3.0 V and 4.0 V at different discharge rates. Figure 1 shows the voltage profile of the LFP electrode (solid line) and full battery (dotted line) during charge/discharge process. It is seen that the higher the C-rate, the higher is the polarization furnished by the counter electrode which reduces the capacity. In Figure 2, the discharge capacity [mAh/g] is plotted vs the number of charge/discharge cycles. Series of 10 cycles at a given C-rate was applied to the battery. Each series was followed by a C/10 cycle (green points). A linear fit has been applied to the first series (omitting first two cycles where instability of the system is observed), in order to calculate the degradation rates. High C-rates are seen to affect the discharge capacity, but the capacity is almost completely recovered (green points) and only a limited degradation occurs. Impedance spectroscopy has been also applied to investigate the LFP cathode degradation. Figure 3 shows the imaginary part of the impedance measured at 50% State-of-Charge after each series of cycles. The relative increase in the impedance arc around 1 KHz (assumed to be associated with charge transfer resistance at the LFP particle surfaces) is seen to gradually decrease with increasing number of series. This indicates that more cycles per series is needed to establish a convincing relation between C-rate and degradation. The degradation studies will be coupled with FIB/SEM analysis in order to observe changes in the pore structure or micro cracks that would affect electronic percolation. Figure 4 displays an example of a fresh LFP cathode after FIB cutting. White particles are LFP grains while the black area contains carbon particles and pores, which are difficult to distinguish from each other. Substitution of the epoxy resin with a silicon resin increases the contrast between pores and carbon particles [1] and this will be used in the forthcoming FIB/SEM analysis. References [1] M. Ender et al, Journal of The Electrochemical Society, 159 (7) A972-A980 (2012) [Formula]
Effect of Ru/CGO versus Ni/CGO Co-Infiltration on the Performance and Stability of STN-Based SOFCs

Electrolyte supported cells (ESC), with Sc2O3-stabilized ZrO2 (ScSZ) electrolytes, Gd-doped ceria (CGO) or M/CGO (M = Ni, Ru) infiltrated Sr0.94Ti0.9Nb0.1O3 (STN94) anodes and LSM/YSZ cathodes, were evaluated for their initial performance and long-term stability. Power density for the Ru/CGO infiltrated cell reached ~0.7 W cm⁻² at 850 °C, 4% H₂O/H₂, whereas the Ni/CGO infiltrated cell reached ~0.3 W cm⁻², with the current morphologies and loadings. Operation at 0.125 A cm⁻², 850 °C, feeding 50% H₂O/H₂ to the anode and air to the cathode, for a period >300 h, showed superior stability for the Ru/CGO infiltrated cell, with ~0.04 mV h⁻¹ degradation rate, when compared to the Ni/CGO infiltrated cell (~0.5 mV h⁻¹). For the Ni/CGO case, the observed degradation has been tentatively linked to initial changes in the electrochemical active area and long-term detrimental interactions between components.

General information
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Organisations: Department of Energy Conversion and Storage, Fundamental Electrochemistry, Ceramic Engineering & Science, Imaging and Structural Analysis
Contributors: Ramos, T., Veltzé, S., Sudireddy, B. R., Jørgensen, P. S., Kuhn, L. T., Holtappels, P.
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Web of Science (2018): Indexed yes
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Scopus rating (2017): CiteScore 1.88 SJR 0.559 SNIP 0.748
Web of Science (2017): Impact factor 2.149
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 1.79 SJR 0.495 SNIP 0.603
Web of Science (2016): Impact factor 1.706
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 2.02 SJR 0.685 SNIP 0.779
Web of Science (2015): Impact factor 1.769
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 2.05 SJR 0.615 SNIP 0.792
Web of Science (2014): Impact factor 2.08
Web of Science (2014): Indexed yes
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 1.99 SJR 0.835 SNIP 0.833
Web of Science (2013): Impact factor 1.546
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
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Web of Science (2012): Impact factor 2.364
Formulation of desert rose structures in vacuum plasma sprayed electrodes for alkaline electrolysis

General information
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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Deutsches Zentrum Für Luft- und Raumfahrt
Contributors: Bentzen, J. J., Zhang, W., Jørgensen, P. S., Bowen, J. R., Reissner, R.
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Publication date: 2014
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Event: Poster session presented at 18th International Microscopy Congress 2014, Prague, Czech Republic.

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Formulation of desert rose structures in vacuum plasma sprayed electrodes for alkaline electrolysis

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In situ characterization of delamination and crack growth of a CGO–LSM multi-layer ceramic sample investigated by X-ray tomographic microscopy

The densification, delamination and crack growth behavior in a Ce0.9Gd0.1O1.95 (CGO) and (La0.85Sr0.15)0.9MnO3 (LSM) multi-layer ceramic sample was studied using in situ X-ray tomographic microscopy (microtomography) to investigate the critical dynamics of crack propagation and delamination in a multilayered sample. Naturally occurring defects, caused by the sample preparation process, are shown not to be critical in sample degradation. Instead defects are nucleated during the debinding step. Crack growth is significantly faster along the material layers than perpendicular to them, and crack growth and delamination only accelerates when sintering occurs.

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Web of Science (2017): Impact factor 3.794
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 3.25 SJR 1.142 SNIP 1.888
Web of Science (2016): Impact factor 3.454
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 3.03 SJR 1.135 SNIP 1.817
Web of Science (2015): Impact factor 2.933
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 3.16 SJR 1.163 SNIP 2.083
Web of Science (2014): Impact factor 2.947
Web of Science (2014): Indexed yes
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Scopus rating (2013): CiteScore 2.57 SJR 1.111 SNIP 1.79
Web of Science (2013): Impact factor 2.307
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
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**General information**

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**Organisations:** Department of Energy Conversion and Storage, Imaging and Structural Analysis, Ceramic Engineering & Science, Fundamental Electrochemistry

**Instability and growth of nanoscale Ce_{0.8}Gd_{0.2}O_{1.9}/NiO infiltrate in Sr_{0.94}Ti_{0.9}Nb_{0.1}O_{3-Zr_{0.84}Y_{0.16}}O_{1.92} anodes for solid oxide fuel cells**

Microstructural evolution of Ce_{0.8}Gd_{0.2}O_{1.9}/NiO (CGO/NiO) co-infiltrated nanoparticles in Sr_{0.94}Ti_{0.9}Nb_{0.1}O_{3-Zr_{0.84}Y_{0.16}}O_{1.92} (STN94-YSZ) anodes for solid oxide fuel cells (SOFCs) is investigated during electrochemical testing in a symmetric cell setup. The CGO/NiO infiltrated symmetric cells were subjected to varying atmospheres of H_{2}O/H_{2} between 650 and 850°C and characterized by electrochemical impedance spectroscopy. Analytical high resolution transmission electron microscopy showed that the CGO/NiO infiltrate was found to coalesce and grow from an indistinguishable CGO/NiO fluorite structure of an average diameter of 5 nm to individual well-connected, but phase-separated, CGO and Ni particles of 50 nm in average. This study confirms that instability and growth of CGO/NiO infiltrates in STN-based SOFC electrodes affect the morphology and can potentially be linked to reported losses in electrochemical performance. © 2014 Elsevier B.V. All rights reserved.
Investigation of nano scaled electro catalysts in ceramic solid oxide fuel cell anodes based on niobium modified strontium titanate

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Organisations: Department of Energy Conversion and Storage, Fundamental Electrochemistry, Ceramic Engineering & Science, Imaging and Structural Analysis
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Source: PublicationPreSubmission
Source-ID: 103528695
Research output: Research - peer-review › Poster – Annual report year: 2014

Micromechanical Modeling of Solid Oxide Fuel Cell Anode Supports based on Three-dimensional Reconstructions
The efficiency and lifetime of solid oxide fuel cells (SOFCs) is compromised by mechanical failure of cells in the system. Improving the mechanical reliability is a major step in ensuring feasibility of the technology. To quantify the stress in a cell, mechanical properties of the different layers need to be accurately known. Since the mechanical properties are heavily dependent on the microstructures of the materials, it is highly advantageous to understand the impact of microstructures and to be able to determine accurate effective mechanical properties for cell or stack scale analyses. The purpose of this work is to provide such a link. State-of-the-art SOFCs are supported by a porous layer of Ni-3YSZ which has a complex microstructure and a drastic difference in behaviors between their phases. This work investigates the microscopic stress distribution and macroscopic creep rate of porous Ni-3YSZ in the operating temperature through numerical micromechanical modeling. Three-dimensional microstructures of Ni-3YSZ anode supports are reconstructed from a two-dimensional image stack obtained via focused ion beam tomography. Time-dependent stress distributions in the microscopic scale are computed by the finite element method. The macroscopic creep response of the porous anode support is determined based on homogenization theory. It is shown that micromechanical modeling provides an effective tool to study the effect of microstructures on the macroscopic properties.

General information
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Organisations: Department of Energy Conversion and Storage, Mixed Conductors, Imaging and Structural Analysis
Contributors: Kwok, K., Jørgensen, P. S., Frandsen, H. L.
Number of pages: 10
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Microstructure analysis of vacuum plasma sprayed electrodes for alkaline electrolysis

General information
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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis
Contributors: Bowen, J. R., Bentzen, J. J., Jørgensen, P. S., Zhang, W.
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Source-ID: 103524991
Research output: Research - peer-review › Conference abstract in proceedings – Annual report year: 2014

On the accuracy of triple phase boundary lengths calculated from tomographic image data
The triple phase boundary (TPB) length is one of the most important quantities obtainable from three dimensional reconstructions of solid oxide fuel cells that utilize porous composite electrodes. However, the choice of TPB calculation method and the voxelation of the microstructures can lead to systematic errors in TPB estimates. Here, two approaches for calculating the TPB density are compared to investigate how different TPB aspects such as curvature, orientation, and phase contact angles affect the results. The first approach applies a correction factor to the TPB length calculated by simply summing voxel (volume element) edge lengths that are shared between voxels of three different phases. The second approach applies a smoothing technique to the TPB curves. The two methods are compared by calculations on different kinds of artificially generated microstructures and on a real SOFC electrode microstructure obtained by focused ion beam tomography. Results are presented showing how specific aspects of different microstructures affect the TPB length calculation error.

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Organisations: Department of Energy Conversion and Storage, Imaging and Structural Analysis, Northwestern University
Contributors: Jørgensen, P. S., Yakal-Kremski, K., Wilson, J., Bowen, J. R., Barnett, S.
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Scopus rating (2017): CiteScore 7 SJR 2.202 SNIP 1.536
Web of Science (2017): Impact factor 6.945
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 6.22 SJR 1.944 SNIP 1.5
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 6.34 SJR 1.9 SNIP 1.667
Site specific 3D pathway analysis of functional energy materials
Long-Term Stability of LSM-YSZ Based Cathodes

A transmission line based model was successfully applied to study the ageing effect in LSM-YSZ cathodes after being exposed to humidified air at 900 °C for up to 3000 h. A decrease in the YSZ conductivity was correlated with the formation of the less conducting monoclinic zirconia. The amount of La2Zr2O7, present in the non-aged samples, decreases with ageing time increasing the number of active sites for charge transfer between LSM and YSZ as well as the LSM conductivity values.
Performance of Electrolyte Supported Solid Oxide Fuel Cells with STN Anodes

In order to replace the state of the art Ni-cermet as SOFC anode, electrolyte supported cells comprising CGO/Ni infiltrated Nb-doped SrTiO3 anodes, and LSM/YSZ cathodes have been developed and tested as single 5 x 5 cm2 cells. The initial performance reached 0.4 W/cm2 at 850 C. Further tests under high fuel utilization and redox cycling have been performed to identify the performance limiting parameters in this new type of full ceramic SOFCs. Measured performances and stability have been further tentatively linked to modifications of the nano-sized infiltrates within the anode.

General information
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Contributors: Veltzé, S., Reddy Sudireddy, B., Jørgensen, P. S., Zhang, W., Kuhn, L. T., Holtappels, P., Ramos, T.
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Web of Science (2017): Indexed yes
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Scopus rating (2016): CiteScore 0.4 SJR 0.228 SNIP 0.253
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 0.36 SJR 0.211 SNIP 0.244
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 0.36 SJR 0.212 SNIP 0.234
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 0.27 SJR 0.192 SNIP 0.231
ISI indexed (2013): ISI indexed no
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 0.29 SJR 0.241 SNIP 0.26
ISI indexed (2012): ISI indexed no
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 0.36 SJR 0.261 SNIP 0.28
ISI indexed (2011): ISI indexed no
BFI (2010): BFI-level 1
Scopus rating (2010): SJR 0.249 SNIP 0.251
BFI (2009): BFI-level 1
Scopus rating (2009): SJR 0.242 SNIP 0.27
BFI (2008): BFI-level 1
Scopus rating (2008): SJR 0.254 SNIP 0.255
Transmission Electron Microscopy Specimen Preparation Method for Multiphase Porous Functional Ceramics

An optimum method is proposed to prepare thin foil transmission electron microscopy (TEM) lamellae of multiphase porous functional ceramics: prefilling the pore space of these materials with an epoxy resin prior to focused ion beam milling. Several advantages of epoxy impregnation are demonstrated by successful preparation of TEM specimens that maintain the structural integrity of the entire lamella. Feasibility of the TEM alignment procedure is demonstrated, and ideal TEM analyses are illustrated on solid oxide fuel cell and solid oxide electrolysis cell materials. Some potential drawbacks of the TEM specimen preparation method are listed for other samples.
Durable and Robust Solid Oxide Fuel Cells

The solid oxide fuel cell (SOFC) is an attractive technology for the generation of electricity with high efficiency and low emissions. Risø DTU (now DTU Energy Conversion) works closely together with Topsoe Fuel Cell A/S in their effort to bring competitive SOFC systems to the market. This 2-year project had as one of its’ overarching goals to improve durability and robustness of the Danish solid oxide fuel cells. The project focus was on cells and cell components suitable for SOFC operation in the temperature range 600 – 750 °C. The cells developed and/or studied in this project are intended for use within the CHP (Combined Heat and Power) market segment with stationary power plants in the range 1 – 250 kWe in mind. Lowered operation temperature is considered a good way to improve the stack durability since corrosion of the interconnect plates in a stack is lifetime limiting at T > 750 °C. The fact that degradation and robustness is not very well explored or understood at operating temperatures below 750 °C, provides motivation for focussing on materials and cells suitable for, and operated in this temperature range.

A significant part of this project was concerned with improved understanding of degradation and failure mechanisms. Improved understanding of performance and lifetime limiting factors will make it possible to develop strategies for counteracting degradation and improving the power density of SOFC based systems, both necessary to advance towards the goals set out in the national plan for SOFC implementation.

General information

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Publication date: 2012
Microstructural evolution of nanosized Ce$_{0.8}$Gd$_{0.2}$O$_{1.9}$/Ni infiltrate in a Zr$_{0.84}$Y$_{0.16}$O$_{1.92}$-Sr$_{0.94}$Ti$_{0.9}$Nb$_{0.1}$O$_{3-\delta}$ based SOFC anode under electrochemical evaluation

CeO$_2$-based materials have received intensive attention as they have a lot of important physical, chemical and electrochemical properties [1]. Recently, Gd-doped CeO$_2$ (CGO)/Ni infiltrate was found to be an effective electrocatalyst, greatly enhancing the electrocatalytic activity for fuel oxidation in solid oxide fuel cells (SOFCs) [2,3].

How stable is the structure of infiltrated nano-sized electrocatalysts under electrochemical operation? This issue is usually addressed by evaluating electrode performance without detailed structural investigations. However, the behavior of electrocatalysts are of paramount importance for performance and performance stability. Therefore an accurate understanding of the microstructure evolution during electrochemical operation will facilitate evaluating performances of SOFC anodes, and in turn optimize its design.

Here we report a wealth of microstructural investigations of Ce$_{0.8}$Gd$_{0.2}$O$_{1.9}$/Ni (hereafter CGO/Ni)-infiltrated Zr$_{0.84}$Y$_{0.16}$O$_{1.92}$ composited Sr$_{0.94}$Ti$_{0.9}$Nb$_{0.1}$O$_{3-\delta}$ (STN94/8YSZ) anode in a symmetric cell design under a short electrochemical evaluation test (fingerprint test), applying electrochemical impedance spectroscopy (EIS) at mild 3% H$_2$O/H$_2$ and harsh 50% H$_2$O/H$_2$ environment at temperature up to 850 °C.

**General information**

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Research output: Research » Poster – Annual report year: 2012

**Multilayer tape cast SOFC – Effect of anode sintering temperature**

Multilayer tape casting (MTC) is considered a promising, cost-efficient, up-scalable shaping process for production of planar anode supported solid oxide fuel cells (SOFC). Multilayer tape casting of the three layers comprising the half cell (anode support/active anode/electrolyte) can potentially be cost-efficient and simplify the half-cell manufacturing process. Fewer sintering steps (co-sintering), as well as fewer handling efforts, will be advantageous for up-scaled production. Previous reports have shown that our laboratory produces mechanically strong, high performing anode supported SOFC, with high reproducibility, by tape casting of the anode support [1]. Recent initial results obtained on SOFC with half-cells produced by successive tape casting (MTC) of anode support, anode and electrolyte layers, followed by cosintering of the half-cell, showed increased performance and stability upon FC operation compared to SOFC with half-cells produced by tape casting of anode support but spraying of active anode and electrolyte [2]. These results have initiated further work on MTC half cells. Initial MTC production results have shown that it is possible to co-sinter the MTC anode half cells in a rather large “temperature-window”. To increase our understanding of the MTC process, obtained microstructures and the resulting electrochemical performance of these SOFC, we here report a study of MTC based cells. The half-cells have been produced and co-sintered at 5 different temperatures from 1255 °C to 1335 °C. This study investigates the effect of the sintering temperature on the anode microstructure analysed via electron microscopy images; and correlate it with electrochemical performance of the anode obtained from full cell testing and analysed via iV-curves and impedance spectroscopy.

**General information**

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Organisations: Department of Energy Conversion and Storage, Applied Electrochemistry, Ceramic Engineering & Science, Imaging and Structural Analysis
Performance-Microstructure Relations in Ni/CGO Infiltrated Nb-doped SrTiO3 SOFC Anodes

Nb-doped SrTiO3 solid oxide fuel cell (SOFC) anodes, infiltrated with CGO/Ni, were investigated by electrochemical impedance spectroscopy (EIS) and high resolution microscopy techniques, upon varying production and testing parameters. The electrochemical analysis involved a combination of distribution of relaxation times (DRT) and complex non-linear least squares (CNLS) fitting routine. These electrodes were studied as singlephase or as composites with 8YSZ. Sr0.94Ti0.9Nb0.1O3-δ/10 vol.% 8YSZ composite infiltrated electrodes were the best overall performers, with enhanced performance stability under higher steam contents. Combined DRT/CNLS showed the existence of at least three contributing processes to the total polarisation resistance (Rp) of these electrodes. Microstructural changes involving infiltrate growth and redistribution were also confirmed.

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Scopus rating (2017): CiteScore 0.44 SJR 0.225 SNIP 0.252
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 0.4 SJR 0.228 SNIP 0.253
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 0.36 SJR 0.211 SNIP 0.244
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 0.36 SJR 0.212 SNIP 0.234
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 0.27 SJR 0.192 SNIP 0.231
ISI indexed (2013): ISI indexed no
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 0.29 SJR 0.241 SNIP 0.26
ISI indexed (2012): ISI indexed no
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 0.36 SJR 0.261 SNIP 0.28
ISI indexed (2011): ISI indexed no
BFI (2010): BFI-level 1
Scopus rating (2010): SJR 0.249 SNIP 0.251
BFI (2009): BFI-level 1
Scopus rating (2009): SJR 0.242 SNIP 0.27
BFI (2008): BFI-level 1
Scopus rating (2008): SJR 0.254 SNIP 0.255
Scopus rating (2007): SJR 0.213 SNIP 0.206
Determination of Three Dimensional Microstructure Parameters from a Solid Oxide Ni/YSZ Electrode after Electrolysis Operation

The interface structure of a Ni/YSZ electrode tested for 1300 hours in steam electrolysis mode is analyzed. We break down the electrode interface structure measurements into total phase interfaces (e.g. total pore surface area), two-phase interfaces (e.g. Ni/YSZ interface area) and triple-phase boundaries and further analyze these based on their percolation to electron, ion and gas sources. The measurements are compared to previous related work. ©2011 COPYRIGHT ECS - The Electrochemical Society

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Organisations: Microstructures and Interfaces, Fuel Cells and Solid State Chemistry Division, Risø National Laboratory for Sustainable Energy
Contributors: Jørgensen, P. S., Bowen, J. R.
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BFI (2017): BFI-level 1
Scopus rating (2017): CiteScore 0.44 SJR 0.225 SNIP 0.252
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 0.4 SJR 0.228 SNIP 0.253
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 0.36 SJR 0.211 SNIP 0.244
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 0.36 SJR 0.212 SNIP 0.234
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 0.27 SJR 0.192 SNIP 0.231
ISI indexed (2013): ISI indexed no
BFI (2012): BFI-level 1
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ISI indexed (2012): ISI indexed no
BFI (2011): BFI-level 1
Scopus rating (2011): CiteScore 0.36 SJR 0.261 SNIP 0.28
ISI indexed (2011): ISI indexed no
BFI (2010): BFI-level 1
Scopus rating (2010): SJR 0.249 SNIP 0.251
BFI (2009): BFI-level 1
Scopus rating (2009): SJR 0.242 SNIP 0.27
BFI (2008): BFI-level 1
Scopus rating (2008): SJR 0.254 SNIP 0.255
Scopus rating (2007): SJR 0.213 SNIP 0.206
Scopus rating (2006): SJR 0.134 SNIP 0.073
Geometrical characterization of interconnected phase networks in three dimensions

In electrochemical devices such as fuel cells or batteries, the microstructure is a determining factor for the performance of the device. To be able to optimize the microstructure, it is important to be able to quantitatively measure key structural parameters, such that systematic studies can be made. We present several general methods for quantitative characterization of network structures without prior assumptions of shape or application. The characterization is performed by extracting distributions of values rather than single value descriptions, thus allowing more detailed comparisons between samples to be made. The methods characterize tortuosity, path diameters, the novel dead ends property and a particle shape independent alternative to a particle size distribution. The parameters are calculated by the computation of arrival time maps by the fast marching method. The methods are applied to the analysis of each of the three phases in a solid oxide fuel cell sample.

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Organisations: Microstructures and Interfaces, Fuel Cells and Solid State Chemistry Division, Risø National Laboratory for Sustainable Energy, Image Analysis and Computer Graphics, Department of Informatics and Mathematical Modeling
Contributors: Jørgensen, P. S., Hansen, K. V., Larsen, R., Bowen, J. R.
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Web of Science (2018): Indexed yes
BFI (2017): BFI-level 1
Scopus rating (2017): CiteScore 1.85 SJR 0.728 SNIP 0.94
Web of Science (2017): Impact factor 1.693
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 1.9 SJR 0.746 SNIP 0.841
Web of Science (2016): Impact factor 1.692
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 2.37 SJR 0.962 SNIP 1.095
Web of Science (2015): Impact factor 2.136
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 2.41 SJR 1.067 SNIP 1.339
Web of Science (2014): Impact factor 2.331
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 1.96 SJR 0.749 SNIP 1.051
Web of Science (2013): Impact factor 2.15
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 1.84 SJR 0.764 SNIP 1.276
Web of Science (2012): Impact factor 1.633
ISI indexed (2012): ISI indexed yes
Ni/YSZ anode – Effect of pre-treatments on cell degradation and microstructures
Anode supported (Ni/YSZ–YSZ–LSM/YSZ) solid oxide fuel cells were tested and the degradation over hundreds of hours was monitored and analyzed by impedance spectroscopy. Test conditions were chosen to focus on the Ni/YSZ anode degradation and all tests were operated at 750°C, a current density of 0.75Acm−2. Oxygen was supplied to the cathode and the anode inlet gas mixture had a high p(H2O)/p(H2) ratio of 0.4/0.6. commercially available gases were applied. The effect of different types of pre-treatments on the Ni/YSZ electrode degradation during subsequent fuel cell testing was investigated. Pre-treatments included operating at OCV (4% and 40% H2O in H2) prior to fuel cell testing, cleaning of the inlet H2 gas at 700°C and processing the anode half cell via multilayer tape casting. Analyses of impedance spectra showed that the increase in the charge transfer reaction resistance in the Ni/YSZ electrode degradation during subsequent fuel cell testing was investigated. Pre-treatments included operating at OCV (4% and 40% H2O in H2) prior to fuel cell testing, cleaning of the inlet H2 gas at 700°C and processing the anode half cell via multilayer tape casting. Analyses of impedance spectra showed that the increase in the charge transfer reaction resistance in the Ni/YSZ (RNi,TPB) was decreased to ¼ or less for the pre-treated and fuel cell tested cells when compared with a non-pre-treated reference tested cell; all operated at the same fuel cell test conditions. Scanning electron microscopy and image analyses for the non-pre-treated reference tested cell and selected pre-treated cells showed significant differences in the area fractions of percolating nickel both in the active anode and support layer.
Quantitative data analysis methods for 3D microstructure characterization of Solid Oxide Cells

The performance of electrochemical ceramic devices such as solid oxide fuel and electrolyzer cells depends on the distribution of constituent phases on the micro or nano scale, also known as the microstructure. The microstructure governs key properties such as ion, electron and gas transport through percolating networks and reaction rates at the triple phase boundaries. Quantitative analysis of microstructure is thus important both in research and development of optimal microstructure design and fabrication. Three dimensional microstructure characterization in particular holds great promise for gaining further fundamental understanding of how microstructure affects performance. In this work, methods for automatic 3D characterization of microstructure are studied: from the acquisition of 3D image data by focused ion beam tomography to the extraction of quantitative measures that characterize the microstructure. The methods are exemplified by the analysis of Ni-YSZ and LSC-CGO electrode samples. Automatic methods for preprocessing the raw 3D image data are developed. The preprocessing steps correct for errors introduced by the image acquisition by the focused ion beam serial sectioning. Alignment of the individual image slices is performed by automatic detection of fiducial marks. Uneven illumination is corrected by fitting hypersurfaces to the spatial intensity variation in the 3D image data. Routine use of quantitative three dimensional analysis of microstructure is generally restricted by the time consuming task of manually delineating structures within each image slice or the quality of manual and automatic segmentation schemes. To solve this, a framework for the automatic segmentation of 3D image data is developed. The technique is based on a level set method and uses numerical approximations to partial differential equations to evolve a 3D surface to capture the phase boundaries. Vector fields derived from the experimentally acquired data are used as the driving forces. The framework performs the segmentation in 3D rather than on a slice by slice basis. It naturally supplies sub-voxel accuracy of segmented surfaces and allows constraints on the surface curvature to enforce a smooth surface in the segmentation. A high accuracy method is developed for calculating two phase boundary surface areas and triple phase boundary length of triple phase systems. The calculations are based on sub-voxel accuracy segmentations of the constituent phases. The method performs a three phase polygonization of the interface boundaries which results in a non-manifold mesh of connected faces. The triple phase boundaries can be extracted from the mesh as connected curve loops without branches. The accuracy of the method is analyzed by calculations on geometrical primitives. A suite of methods is developed for characterizing the shape and connectivity of phase networks. The methods utilize the fast marching method to compute distance maps and optimal paths in the microstructure network. The extracted measurements are suited for the quantitative comparison and evaluation of microstructures. The quantitative measures characterize properties of network path tortuosity, network thickness, transport path width and dead ends.

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A framework for automatic segmentation in three dimensions of microstructural tomography data

Routine use of quantitative three dimensional analysis of material microstructure by in particular, focused ion beam (FIB) serial sectioning is generally restricted by the time consuming task of manually delineating structures within each image slice or the quality of manual and automatic segmentation schemes. We present here a framework for performing automatic segmentation of complex microstructures using a level set method. The technique is based on numerical approximations to partial differential equations to evolve a 3D surface to capture the phase boundaries. Vector fields derived from the experimentally acquired data are used as the driving forces. The framework performs the segmentation in 3D rather than on a slice by slice basis. It naturally supplies sub-voxel precision of segmented surfaces and allows constraints on the surface curvature to enforce a smooth surface in the segmentation. Two applications of the framework are illustrated using solid oxide cell materials as examples.

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Web of Science (2018): Indexed yes
BFI (2017): BFI-level 1
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Web of Science (2017): Impact factor 2.929
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 1
Scopus rating (2016): CiteScore 2.82 SJR 1.896 SNIP 1.176
Web of Science (2016): Impact factor 2.843
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 1
Scopus rating (2015): CiteScore 2.78 SJR 2.066 SNIP 1.326
Web of Science (2015): Impact factor 2.874
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 1
Scopus rating (2014): CiteScore 2.59 SJR 1.628 SNIP 1.598
Web of Science (2014): Impact factor 2.436
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 2.66 SJR 1.761 SNIP 1.323
Web of Science (2013): Impact factor 2.745
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes
BFI (2012): BFI-level 1
Scopus rating (2012): CiteScore 2.31 SJR 1.866 SNIP 1.562
Web of Science (2012): Impact factor 2.47
ISI indexed (2012): ISI indexed yes
Web of Science (2012): Indexed yes
BFI (2011): BFI-level 1
Auto-Segmentation of Head and Neck Cancer using Textural features

Purpose: The conventional treatment for non-metastatic Head & Neck squamous cell carcinoma (HNSCC) is radiation therapy. Despite technological advances and improved efficacy radiation therapy still relies on manual delineation of gross tumour volume which is both time consuming and prone to inter- and intra observer variability. Several automatic segmentation methods have been developed using positron emission tomography (PET) and/or computerised tomography (CT). The aim of the present study is to develop a model for 3-dimensional auto-segmentation, the level set method, to contour gross tumour volumes (GTV) in a training set of 20 HNSCC patients and evaluate its performance in an independent test set of 25 patients. Materials and Methods: 100 PET/CT textural features were extracted from manual contours of GTV on a training set. The training set consisted of PET and CT scans from 20 patients randomly selected among 45 cases with hypopharyngeal carcinoma treated with radiotherapy. All contours had been performed by experienced radiologists for treatment planning. The Jeffreys-Matusita (JM) distance, a measure of similarity between distributions, was calculated for combinations of features inside and outside the GTV respectively to choose an appropriate feature combination for segmentation of the GTV. The feature combination with the highest dissimilarity was extracted on PET and CT images from the remaining 25 HNC patients. Using these features as input for a level set segmentation method the tumours were segmented automatically. Segmentation results were evaluated against manual contours of radiologists using the DICE coefficient, and sensitivity. The result of the level set approach method was compared with threshold segmentation of PET standard uptake value (SUV) of 3 or 20% of maximal intensity and tested with a paired t-test. Results: The JM analysis determined a combination of 8 textural features as appropriate for segmentation giving a distance of 1.1 out of 1.4. For the level set segmentation the DICE coefficient and sensitivity were 0.48±0.18 (mean ± standard deviation) and 0.57±0.24 respectively. Mean DICE coefficient for the 3 SUV and 20% intensity threshold segmentation were respectively 0.41±0.22 and 0.40±0.22, giving p-values of 0.04 and 0.02 for a higher DICE coefficient from the level set segmentation. For sensitivity the threshold segmentation yielded 0.52±0.24 and
0.51±0.26 for 3SUV and 20% intensity respectively yielding p-values of 0.01 and 0.03. Conclusion: The level set method provides a more robust and stable method for segmentation of HNSCC at hypopharynx than threshold segmentation. But it should be improved in order to resemble the manual contours of radiologist. The segmentation could serve as an initial GTV estimate for manual corrections reducing both time and variance in the process of GTV contouring.

**General information**

State: Published  
Organisations: Department of Informatics and Mathematical Modeling, Image Analysis and Computer Graphics, Department of Electrical Engineering, Biomedical Engineering, Copenhagen University Hospital 
Publication date: 2010  
Peer-reviewed: Yes  
Event: Abstract from 29th European Society for Therapeutic Radiology and Oncology, Barcelona, Spain.  
Keywords: Segmentation, PET/CT, Deformable models, Head & Neck Cancer  
Source: orbit  
Source-ID: 265670  
Research output: Research - peer-review > Conference abstract for conference – Annual report year: 2010

**Characterization of the 3-dimensional microstructure of a graphite negative electrode from a Li-ion battery**

The 3-dimensional microstructure of a porous electrode from a lithium-ion battery has been characterized for the first time. We use X-ray tomography to reconstruct a 43 × 348 × 478 μm sample volume with voxel dimensions of 480 nm, subsequent division of the reconstructed volumes into sub-volumes of different sizes allow us to determine microstructural parameters as a function of sub-division size. We show that the minimum size for a representative volume element is about 43 × 60 × 60 μm for volume-specific surface area, but as large as the full sample volume for porosity and tortuosity.

**General information**

State: Published  
Organisations: Microstructures and Interfaces, Fuel Cells and Solid State Chemistry Division, Risø National Laboratory for Sustainable Energy, Imperial College London, Natural History Museum, General Motors R&D  
Contributors: Shearing, P., Howard, L., Jørgensen, P. S., Brandon, N., Harris, S.  
Pages: 374-377  
Publication date: 2010  
Peer-reviewed: Yes

**Publication information**

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Volume: 12  
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Web of Science (2018): Indexed yes  
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Web of Science (2017): Impact factor 4.66  
Web of Science (2017): Indexed yes  
BFI (2016): BFI-level 1  
Scopus rating (2016): CiteScore 4.53 SJR 1.626 SNIP 1.086  
Web of Science (2016): Impact factor 4.396  
Web of Science (2016): Indexed yes  
BFI (2015): BFI-level 1  
Scopus rating (2015): CiteScore 4.77 SJR 1.628 SNIP 1.176  
Web of Science (2015): Impact factor 4.569  
BFI (2014): BFI-level 1  
Scopus rating (2014): CiteScore 5.16 SJR 1.832 SNIP 1.342  
Web of Science (2014): Impact factor 4.847  
Web of Science (2014): Indexed yes  
BFI (2013): BFI-level 2  
Scopus rating (2013): CiteScore 4.97 SJR 1.808 SNIP 1.422  
Web of Science (2013): Impact factor 4.287
High accuracy interface characterization of three phase material systems in three dimensions
Quantification of interface properties such as two phase boundary area and triple phase boundary length is important in the characterization of many material microstructures, in particular for solid oxide fuel cell electrodes. Three-dimensional images of these microstructures can be obtained by tomography schemes such as focused ion beam serial sectioning or micro-computed tomography. We present a high accuracy method of calculating two phase surface areas and triple phase length of triple phase systems from subvoxel accuracy segmentations of constituent phases. The method performs a three phase polygonization of the interface boundaries which results in a non-manifold mesh of connected faces. We show how the triple phase boundaries can be extracted as connected curve loops without branches. The accuracy of the method is analyzed by calculations on geometrical primitives.

General information
State: Published
Organisations: Microstructures and Interfaces, Fuel Cells and Solid State Chemistry Division, Risø National Laboratory for Sustainable Energy, Image Analysis and Computer Graphics, Department of Informatics and Mathematical Modeling
Contributors: Jørgensen, P. S., Hansen, K. V., Larsen, R., Bowen, J. R.
Pages: 8168-8176
Publication date: 2010
Peer-reviewed: Yes
Towards automated 3D electrode microstructure characterisation from a data analysis perspective

General information
State: Published
Organisations: Fuel Cells and Solid State Chemistry Division, Microstructures and Interfaces, Risø National Laboratory for Sustainable Energy
Contributors: Bowen, J. R., Jørgensen, P. S.
Publication date: 2010

Event information
Event: Workshop on Image Analysis in SOFC Degradation Research
Location: Brussels
Keywords: Materials characterization and modelling, Materials and energy storage

Surface Area Determination of Metal Ceramic Composite by FIB Sectioning and BET Measurements

General information
State: Published
Organisations: Fuel Cells and Solid State Chemistry Division, Microstructures and Interfaces, Risø National Laboratory for Sustainable Energy, Ceramic processing
Pages: 324-325
Publication date: 2009
Peer-reviewed: Yes
Unsupervised Assessment of Subcutaneous and Visceral Fat by MRI

This paper presents a method for unsupervised assessment of visceral and subcutaneous adipose tissue in the abdominal region by MRI. The identification of the subcutaneous and the visceral regions were achieved by dynamic programming constrained by points acquired from an active shape model. The combination of active shape models and dynamic programming provides for a both robust and accurate segmentation. The method features a low number of parameters that give good results over a wide range of values. The unsupervised segmentation was compared with a,
Robust automatic high resolution segmentation of SOFC anode porosity in 3D.
Routine use of 3D characterization of SOFCs by focused ion beam (FIB) serial sectioning is generally restricted by the time consuming task of manually delineating structures within each image slice. We apply advanced image analysis algorithms to automatically segment the porosity phase of an SOFC anode in 3D. The technique is based on numerical approximations to partial differential equations to evolve a 3D surface to the desired phase boundary. Vector fields derived from the experimentally acquired data are used as the driving force. The automatic segmentation compared to manual delineation reveals and good correspondence and the two approaches are quantitatively compared. It is concluded that the automatic approach is more robust, more reproducible and orders of magnitude quicker than manual segmentation of SOFC anode porosity for subsequent quantitative 3D analysis. Lastly it is anticipated that the methodology can be extrapolated to all phases in the anode.

General information
State: Published
Automatic assessment of intrabdominal fat by MRI

General information
State: Published
Organisations: Image Analysis and Computer Graphics, Department of Informatics and Mathematical Modeling
Contributors: Jørgensen, P. S., Larsen, R., Wraae, K., Erbsbøll, B. K. (ed.)
Publication date: 2007
Peer-reviewed: Yes
Event: Abstract from 2nd Conference on Image Analysis and In-Vivo Pharmacology, Roskilde, Denmark.
Electronic versions:
imm5150.pdf
Source: orbit
Source-ID: 200541
Research output: Research - peer-review » Conference abstract for conference » Annual report year: 2007

Boundary based segmentation of 2D electron microscope images. Ph.D. presentation

General information
State: Published
Organisations: Electrochemistry, Fuel Cells and Solid State Chemistry Division, Risø National Laboratory for Sustainable Energy
Contributors: Jørgensen, P. S.
Publication date: 2007
Peer-reviewed: No
Event: Paper presented at SERC biannual meeting, Risø, Denmark.
Source: orbit
Source-ID: 216098
Research output: Research » Paper » Annual report year: 2007

Projects:

Physical model priors for tomogram segmentation
Brenne, E. O., PhD Student, Department of Energy Conversion and Storage
Jørgensen, P. S., Main Supervisor, Department of Energy Conversion and Storage
Dahl, V. A., Supervisor
Marie Curie (EU-stipendium) m/virksomhed
01/08/2018 → 31/07/2021
Award relations: Physical model priors for tomogram segmentation
Project: PhD

Electrodeposition of Metallic 3D Surface-Profiles for Superconductor Tapes
Master thesis project by Suzanne Zamany Andersen. Thesis abstract: The work in this thesis is based on a recently introduced 3D surface-prole technique, i.e. the two-level undercut-prole substrate (2LUPS) concept [1]-[2], used for production of multi-lametary high-temperature coated conductor (CC) tapes. Reducing the superconductor lament width linearly reduces the alternating current hysteretic energy losses [3], and it enables manufacturing of stable high-temperature superconducting magnets [4]. A new process of tape masking and Ni-based electroplating on a Ni-W metal alloy substrate to form similar 3D surfaceproles as those achieved by the 2LUPS concept [5], which is based on two levels of plateaus connected via an undercut-prole, is investigated. The undercut-prole should be large enough to enable a shading eect during subsequent physical vapor deposition (PVD) of layers, thereby creating self-formed and physically separated superconductor laments on the two plateaus, while still utilizing the full width of the CC. This will theoretically increase the engineering current density compared to current lament techniques utilizing e.g. laser striation or mechanical
scribing. Inspection of the metal substrate cross-section using focused ion beam milling and scanning electron microscopy (FIBSEM) reveals that an undercut-prole is achieved by using kapton tape as a mask while electroplating nickel to create the upper plateaus. The arithmetic surface roughness of the electroplated nickel layer is determined via atomic force microscopy (AFM) to be suitable for CC fabrication. To verify if the undercut-prole is sucient, an electrically insulating layer of SiO, simulating the buer layers in CCs, followed by an electrically conductive layer of Ag, simulating the superconducting layer, is deposited using PVD, and four-point probe measurements to create I/V characteristics are used to measure resistance across plateaus. The plateaus are deemed electrically insulated from each other, as the resistances from each insulating layer adds up to the total resistance through both plateaus. Accordingly, it is expected that these new electroplated 3D surface-proles will also enable lamentization of superconductors produced by PVD processes. A small caveat to these ndings, is the lack of a suitable prole for the use in CC fabrication being manufactured in this project. The adhesive in the masking tape creates bulges or protrusions in the prole, so a further study on thinner adhesive layers or a dierent masking material altogether is needed. The possibility of texture transfer from the Ni-W metal substrates to the plated Ni layer is also investigated, for the use in the cheaper rolling assisted bi-axially textured substrate (RABiTS) fabrication process. The electrodeposited Ni would during annealing at low temperatures experience an abnormal grain growth stage, thereby rendering it incapable of attaining the texture needed for RABiTS fabrication. Furthermore, the thermal grooving during annealing of the pure Ni could also become a problem for the ion beam assisted deposition (IBAD) process, as a surface roughness of

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Andersen, S. Z., Project Participant, Department of Physics, Experimental Surface and Nanomaterials Physics
Jørgensen, P. S., Supervisor, Department of Energy Conversion and Storage, Imaging and Structural Analysis
Nielsen, P. H., Supervisor, Department of Energy Conversion and Storage, Imaging and Structural Analysis
Bentien, A., Examiner, Aarhus University
22/08/2016 → 12/02/2017
Keywords: electrochemistry, electroplating, metal substrates, Coated conductor, Superconductor, topography, EBSD, FIB-SEM, texture
Collaborators: Aarhus University
Project: Research

Dark Field X-ray Microscopy of energy materials
Sierra Trujillo, J. X., PhD Student, Department of Energy Conversion and Storage
Bowen, J. R., Main Supervisor, Department of Energy Conversion and Storage
Jørgensen, P. S., Supervisor, Department of Energy Conversion and Storage
Poulsen, H. F., Supervisor
Hagen, A., Examiner, Department of Energy Conversion and Storage
Sørensen, H. O., Examiner
Villanova, J., Examiner
Samfinansieret - Andet
15/06/2015 → 14/07/2018
Award relations: Dark Field X-ray Microscopy of energy materials
Project: PhD

In-situ 3D microstructure characterisation of solid oxide fuel cells using X-ray tomography methods
De Angelis, S., PhD Student, Department of Energy Conversion and Storage
Bowen, J. R., Main Supervisor, Department of Energy Conversion and Storage
Jørgensen, P. S., Supervisor, Department of Energy Conversion and Storage
Lauridsen, E. M., Supervisor, Department of Energy Conversion and Storage
Andreasen, J. W., Examiner, Department of Energy Conversion and Storage
Carbone, G., Examiner
Chiu, W. K. S., Examiner
Forskningsrådsfinansiering
15/08/2014 → 13/12/2017
Award relations: In-situ 3D microstructure characterisation of solid oxide fuel cells using X-ray tomography methods
Project: PhD

Automatic Quantitative Image Analysis of 3D Micrographs
Jørgensen, P. S., PhD Student, Department of Informatics and Mathematical Modeling
Larsen, R., Main Supervisor, Department of Informatics and Mathematical Modeling
Bowen, J. R., Supervisor, Department of Energy Conversion and Storage, Imaging and Structural Analysis
Hansen, K. V., Supervisor
Børregaard, L. R., Examiner
Forskningsrådsfinansiering
01/03/2007 → 22/09/2010
RESelyser: Hydrogen from RES: pressurised alkaline electrolyser with high efficiency
The project RESelyser develops high pressure, highly efficient, low cost alkaline water electrolyser that can be integrated with renewable energy power sources (RES) using an advanced membrane concept, highly efficient electrodes and a new cell concept. A new concept with a three electrolyte loop system will be developed demonstrating even higher performance than conventional two electrolyte loop systems. This three electrolyte loop system will use a new separator membrane with internal electrolyte circulation and an adapted cell to improve mass transfer, especially gas evacuation. Intermittent and varying load operation connected to an RES will be addressed by improved electrode stability and a cell concept for increasing the gas purity of hydrogen and oxygen especially at low power as well as by a system concept. Electrolysers up to 30 kW with 6 Nm³/h hydrogen production will be realized in the project. The primary pressure of the electrolyser will be above 25 bar (without the use of a compressor) to reduce the power loss for hydrogen compression to a minimum. All components of the system will be analyzed for their costs and developed to reduce the system price such that hydrogen can be produced at 3000 €/(Nm³/h). An extrapolation to a primary electrolyser pressure of 100-150 bar is considered.
Bowen, J. R., Project Manager, Department of Energy Conversion and Storage, Imaging and Structural Analysis
Jørgensen, P. S., Project Participant, Department of Energy Conversion and Storage, Imaging and Structural Analysis, Fuel Cells and Solid State Chemistry Division, Microstructures and Interfaces
Bentzen, J. J., Project Participant, Department of Energy Conversion and Storage, Imaging and Structural Analysis
Project ID: 48099
External Project ID: 278732
FCH JU: DKK727,162.00
01/11/2011 → 30/04/2015
Keywords: Alkaline Electrolysis, Hydrogen
Collaborators: Flemish Institute for Technological Research, Hydrogenics Europe NV, Deutsches Zentrum Für Luft- und Raumfahrt
Award relations: Hydrogen from RES: pressurised alkaline electrolyser with high efficiency
Project: Research

Activities:

Solid oxide fuel cells – from a materials science perspective
Period: 16 May 2011
Peter Stanley Jørgensen (Participant)
Risø National Laboratory for Sustainable Energy
Fuel Cells and Solid State Chemistry Division
Microstructures and Interfaces

Description
Solid oxide fuel cells (SOFCs) efficiently convert chemical energy directly into electricity using fuels ranging from pure hydrogen to diesel. With the use of bio-fuels, SOFC electricity production is CO2neutral. When operated in reverse as solid oxide electrolysis cells, it is possible to store excess electricity from renewable sources in the form of CO2neutral fuels (e.g. for the transport sector) and simultaneously “load balance” the electricity grid. SOFCs are now on the verge of market entry for a variety of heat and power production applications. SOFCs are complex multilayer structures (typically planar). In the electrochemically active layers, a dense ion-conducting ceramic electrolyte separates a porous anode and cathode that are typically electron and ion-conducting composites. The electrolyte becomes an ionic conductor at high temperatures (> 600°C) and the electrodes are exposed to harsh atmospheres during operation. These conditions place stringent materials demands on oxidation resistance, thermal expansion co-efficient matching, creep resistance, mechanical strength, materials compatibility, and electronic and ionic conductivity. The fundamentals of SOFC operation, production, cell-testing and electrode characterisation will be presented in the context of SOFC stacks and systems. Each of these aspects are critically interlinked from a materials science perspective. SOFC design considerations include materials compatibility, production methods, performance, durability, operational requirements, and system cost. Characterisation is an important design step for SOFC performance and durability. Post-mortem investigations commonly use electron microscopy of cell components in combination with cell test data to determine the performance limiting mechanisms. Traditionally, electron microscopy is performed in 2D using scanning and transmission microscopes. Advances in focused ion beam-scanning electron microscopy (FIB-SEM) have allowed nanometre-scale 3D tomographic reconstruction of electrodes, and the possibility to fully describe their stochastic structure and phase percolation. Recent developments in 3D reconstruction methods will be presented in the latter half of the talk.
Documents:
Related event

**Solid oxide fuel cells – from a materials science perspective**
16/05/2011 → 16/05/2011
Risø, Denmark
Activity: Attending an event › Participating in or organising workshops, courses, seminars etc.

**Workshop on Image Analysis in SOFC Degradation Research**
Period: 9 Sep 2010
Peter Stanley Jørgensen (Participant)
Risø National Laboratory for Sustainable Energy
Fuel Cells and Solid State Chemistry Division
Microstructures and Interfaces

**Description**
Towards automated 3D electrode microstructure characterisation from a data analysis perspective: Workshop on Image Analysis in SOFC Degradation Research

Place: Brussels

Related event

**Workshop on Image Analysis in SOFC Degradation Research**
09/09/2010 → 09/09/2010
Brussels
Activity: Attending an event › Participating in or organising workshops, courses, seminars etc.

**Towards automated 3D electrode microstructure characterization from a data analysis perspective**
Period: 6 Jul 2010
Peter Stanley Jørgensen (Speaker)
Risø National Laboratory for Sustainable Energy
Fuel Cells and Solid State Chemistry Division
Microstructures and Interfaces

**Description**
Place: Symposium on solid oxide cell electrodes in 3D, Risø (DK)
Documents:
Stanley Jørgensen_presentation.pdf
Links:
http://www.risoe.dtu.dk/Conferences/3DSOC/Peter%20Stanley%20abstract.aspx (DOC-OA)

Related external organisation

**Unknown external organisation**
Activity: Talks and presentations › Conference presentations