Technical University of Denmark

Microstructure analysis of vacuum plasma sprayed electrodes for alkaline electrolysis

Bowen, Jacob R.; Bentzen, Janet Jonna; Jørgensen, Peter Stanley; Zhang, Wei

Published in:
Conference abstracts - 18th International Microscopy Congress (IMC 2014)

Publication date:
2014

Type of presentation: Oral

MS-14-O-2142 Microstructure analysis of vacuum plasma sprayed electrodes for alkaline electrolysis

Bowen J. R.[1], Bentzen J. J.[1], Jørgensen P. S.[1], Zhang W.[1]

[1]Department of Energy Conversion and Storage, Technical University of Denmark, Risø Campus, Frederiksborgvej 399, 4000 Roskilde

Email of the presenting author: jrbo@dtu.dk

The EU FCH-JU RESelyser project seeks to develop high pressure, high efficiency and low cost alkaline water electrolysers that can operate variably and intermittently to meet the demands for integration into energy networks relying on fluctuating renewable energy. The project utilizes NiAlMo alloy electrodes produced at the German Aerospace Center (DLR) by vacuum plasma spraying (VPS). VPS results in a heterogeneous microstructure consisting of a multitude of intermetallic phase subdomains and pores. We present the results of a broad palette of characterization techniques including SEM, TEM and 3D reconstruction by FIB serial sectioning to analyze this complex structure in the initial state and post mortem.
Electrode surfaces and cross sections are analyzed by high resolution SEM and EDS. The analyses of the cross sections reveal a multitude of complex material structures in the activated electrode (See Figure 1) stemming from the vacuum plasma spraying and electrode activation by leaching of Al and some Al containing intermetallic phases. Desert rose like nano flake structures are observed (See Figure 1) on the electrode surface and in the pores on several electrodes. The desert rose structure is confirmed by TEM to consist primarily of NiO and Al_2NiO_4 like phases (similar lattice parameters). We discuss implications and possible causes of the desert rose structure.
3D reconstructions of the electrodes are made by FIB serial sectioning. The pore space is analyzed in regards to the length, connectivity and tortuosity of the pore transport pathways that allow the KOH electrolyte to infiltrate the porous electrode. Figure 2 shows a reconstructed data cube from an electrode in the initial leached state and the corresponding extracted pore space. The pore space structure is revealed to consist primarily of coarse scale planar like pores parallel to the electrode surface. These thin but coarse pores range in thickness between 2 µm down to some 10s of nanometers at their extremities. In addition, there is a significant pore volume fraction of sub 100 nm wide pores associate with the dissolution of Al from the dentritic morphology of the Raney type NiAl alloy original particles. The combination of the planar pore pathways running parallel to the surface and the fine scale dendritic type pore space significantly complicates the analysis of the 3D pore surface due to the need to reconstruct large volumes at high resolution to both be able to resolve the pores and image the full pathway that connects the pores to the surface of the sample.
The long-term goal is the development of a generic electrode micro/nano structure analysis philosophy for relating electrochemical processes during hydrogen production to highly heterogeneous porous VPS electrodes.

Acknowledgement: The research leading to these results has received funding from the European Union’s Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° [278732] 10.

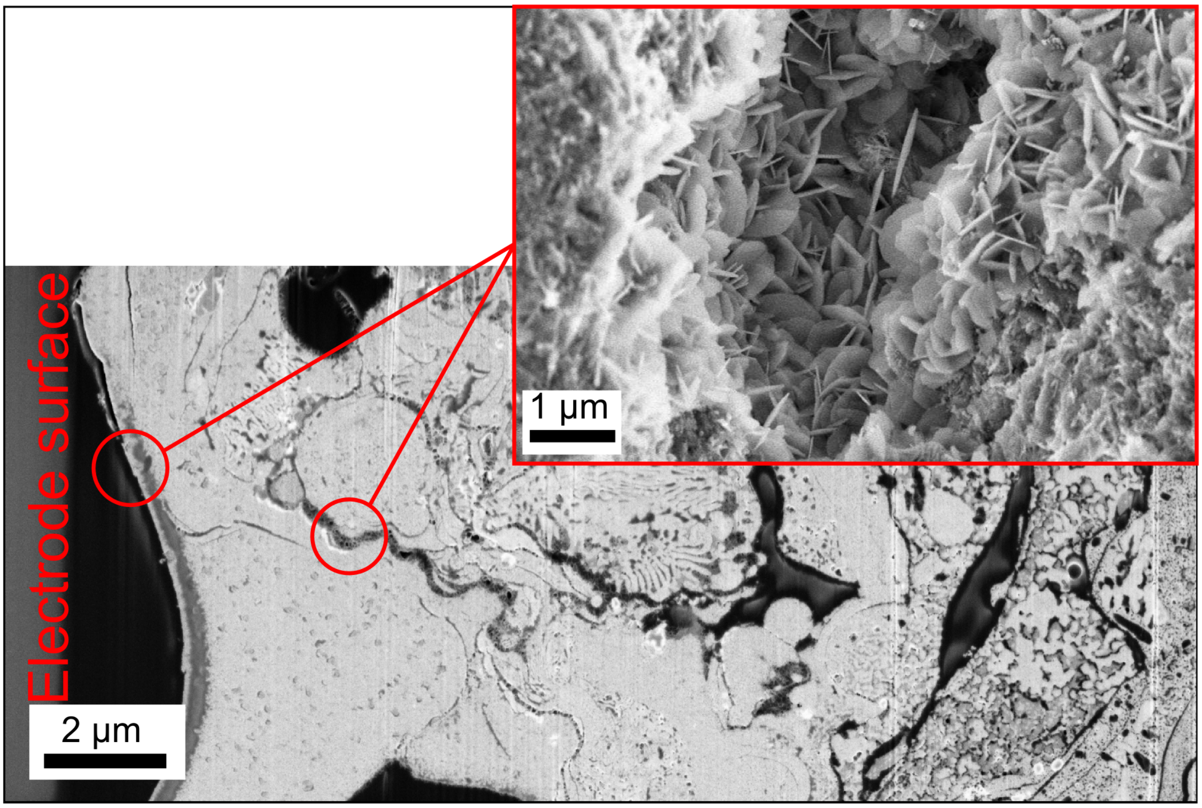

Fig. 1: A FIB cross section of a leached electrode. The surface of the electrode is to the left in the image. The inset shows an SEM surface image of the desert rose like nano flake structures observed on the electrode surface and in the pores on some electrodes.

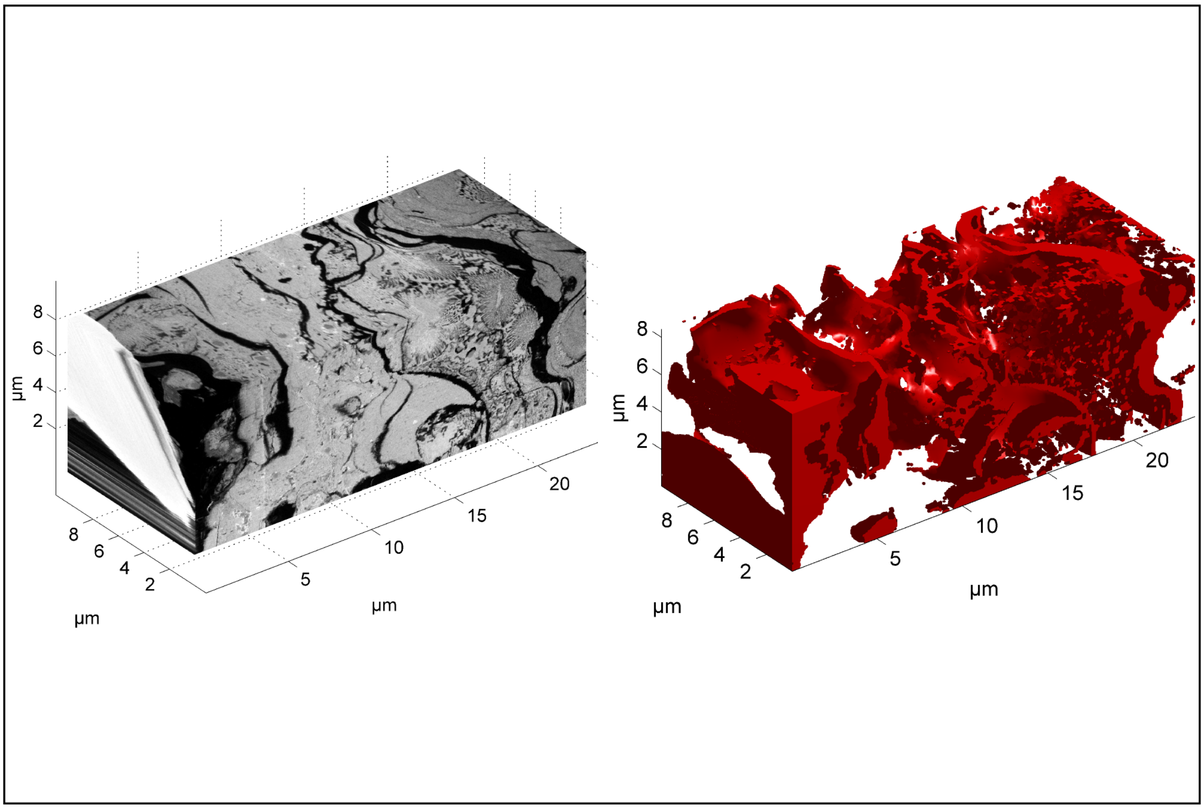

Fig. 2: A visualization of the 3D reconstruction of an electrode by FIB serial sectioning. (left) A visualization of the reconstructed image data. The bright artefact on the left side is the electrode surface. (right) A surface rendering of the pores in the electrode.