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Tracking Electronic Pathways in Energy Materials by Low Voltage Scanning Electron Microscopy

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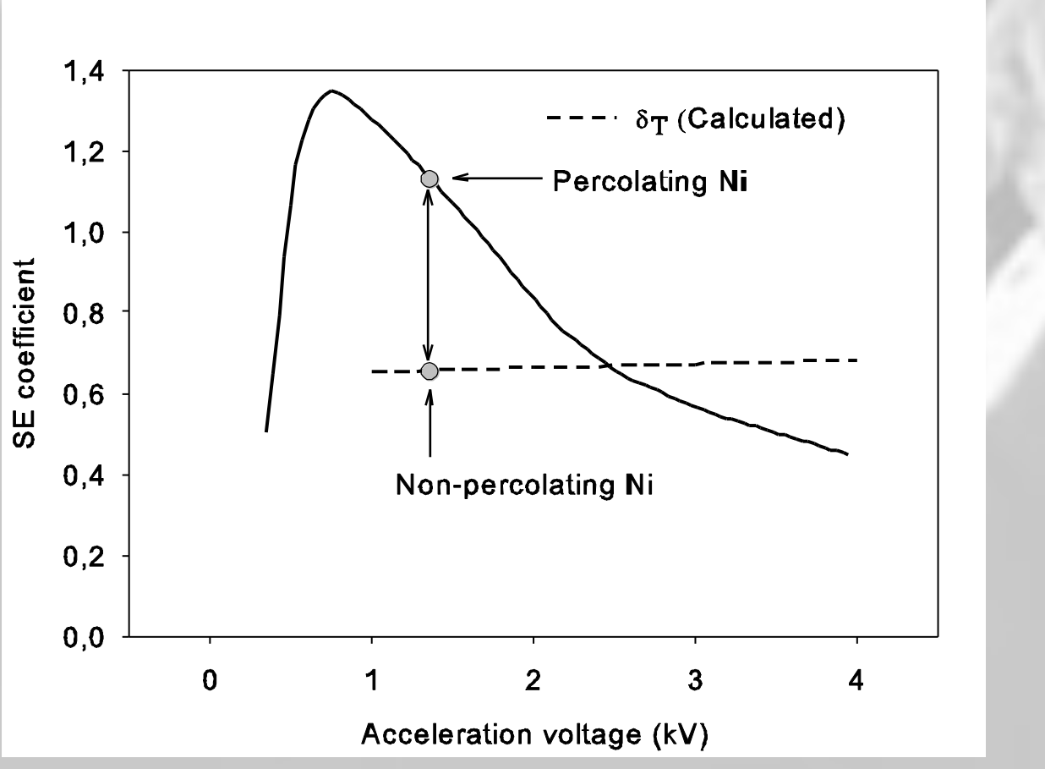
Introduction

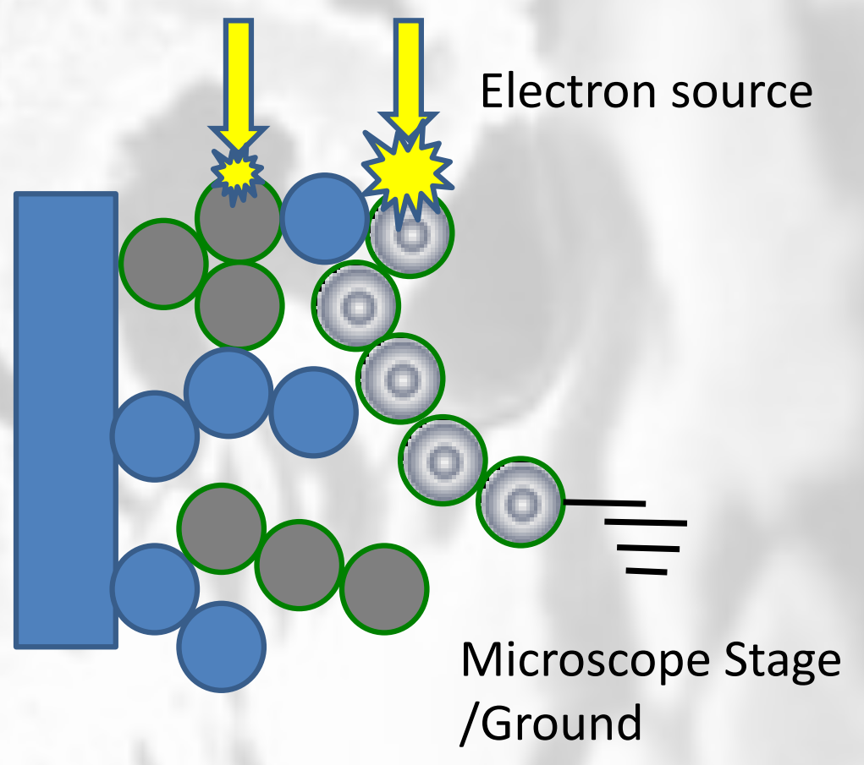
Electrodes for energy conversion devices such as solid oxide fuel cells (SOFC) and electrolysis cells (SOEC) commonly consist of mixtures of electronically and ionically conducting ceramic and metallic materials in order to transport electrons and facilitate charge transfer to/from the electrolyte. For optimal performance it is paramount that the electronically conducting phases are well interconnected throughout the electrodes to create the required electronic connection from the electrolyte to the external circuit. Applying low voltage scanning electron microscopy and surface potential contrast [1] the interconnected electronic pathways within the electrodes can be tracked and evaluated. We here present examples of visualization of electronically percolating phases in electrodes for SOFC, SOEC, and devices for electrochemical reduction of NOx from exhaust gas from vehicles.

Experimental

The microstructural investigations were performed using FEGSEMs Zeiss Supra 35 and Zeiss Merlin applying acceleration voltages in the range 0.9 kV to 1.3 kV. Many materials have a secondary emission coefficient σ higher than 1 in the low voltage range [2]. This is illustrated in the case of Ni in the schematic drawing below. The in-lens secondary detector in a Zeiss Supra 35 microscope mainly picks up the part of the secondary signal that is produced directly by the primary electrons (the SE1 signal); e.g. an in-lens image of an SOFC Ni electrode at 1 kV will therefore show the percolating Ni with an emission coefficient around 1.3 provided that the sample is electrically connected to the stage. Contrary to this, the non-percolating Ni and the YSZ will charge until the barrier for secondary emission is so high that the sum of the backscatter coefficient and σ is 1. They will in other words be imaged with a secondary emission coefficient around 0.7.

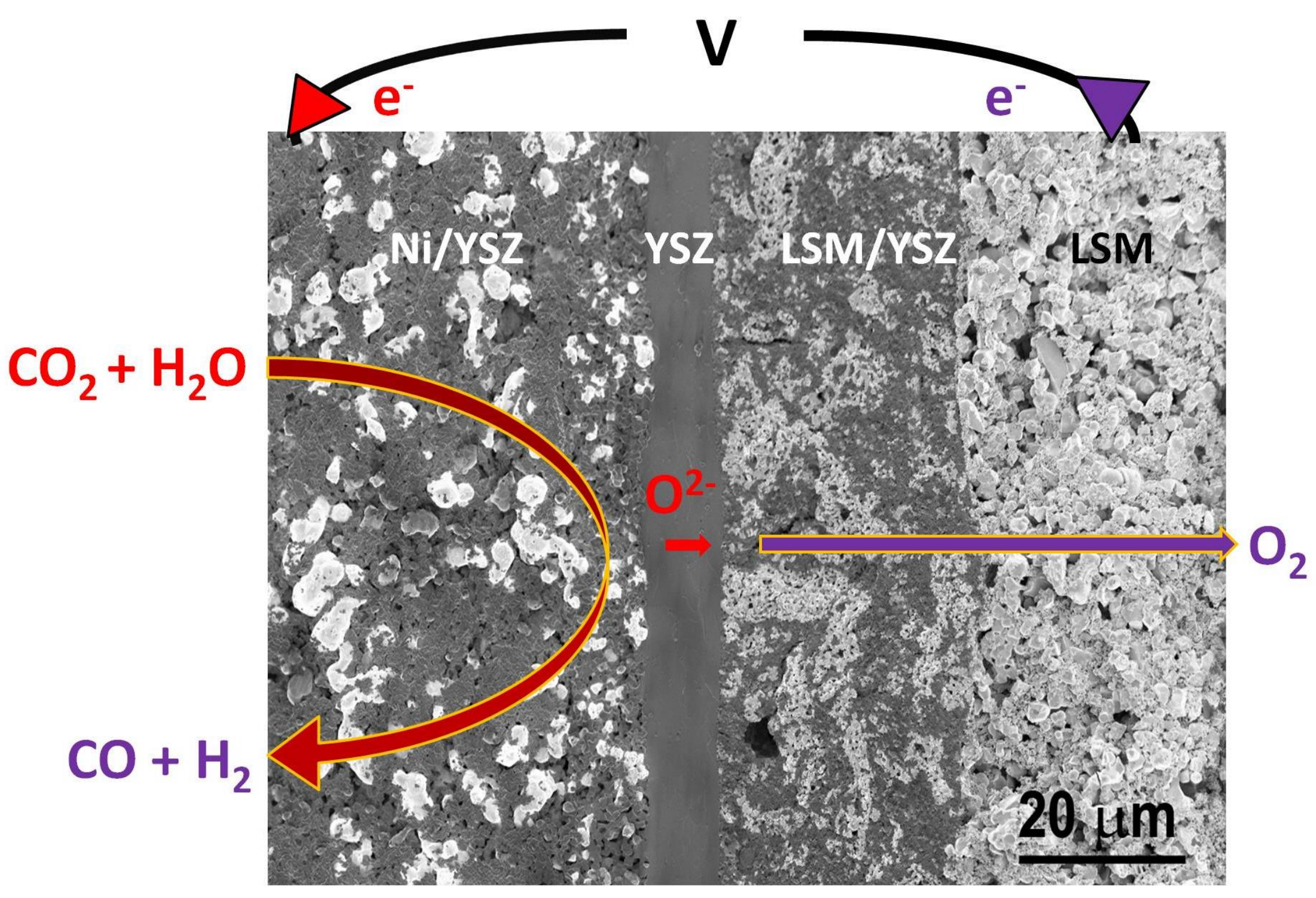


Secondary electron coefficient for Ni as a function of acceleration voltage.

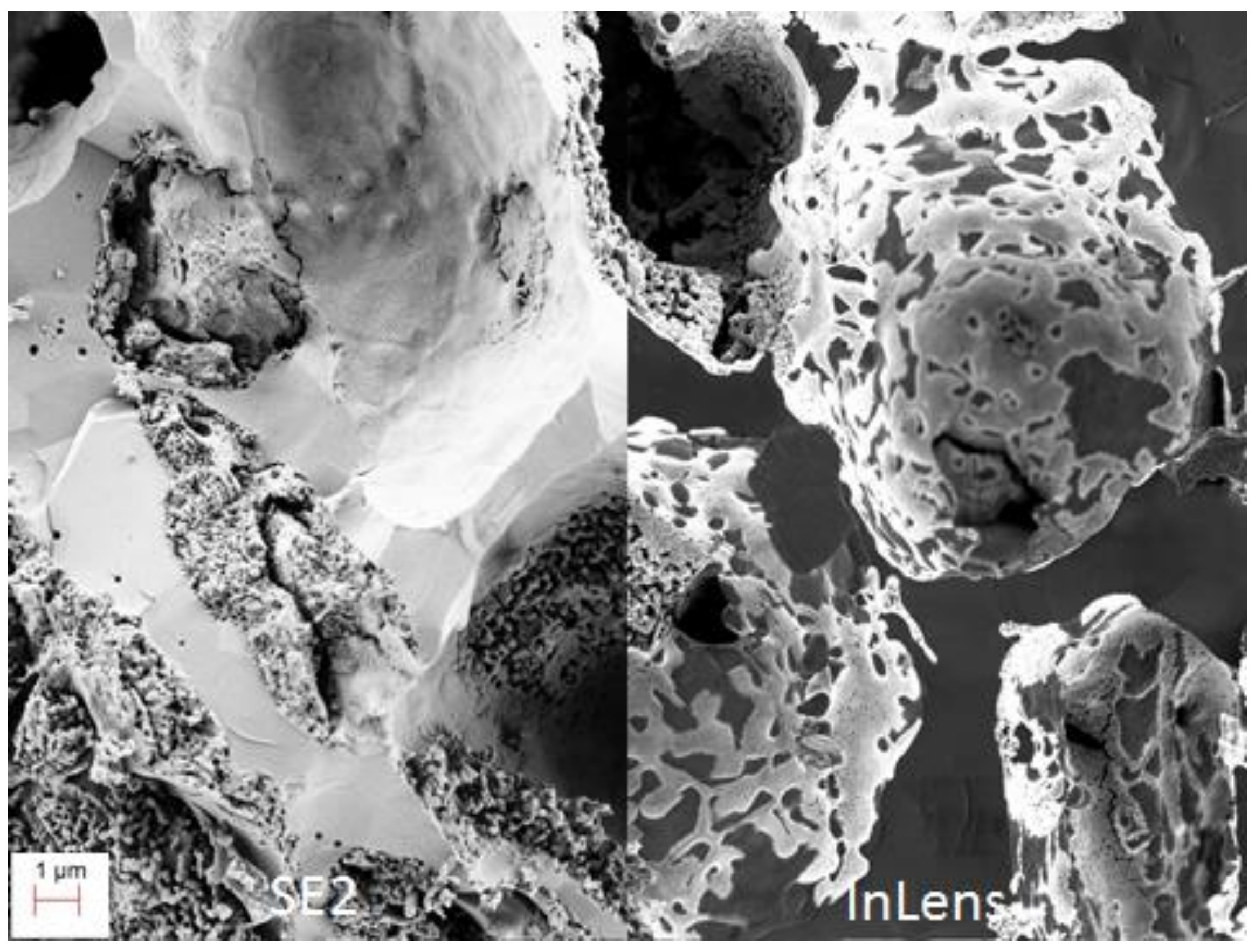


A schematic drawing illustrating the percolation contrast mechanism.

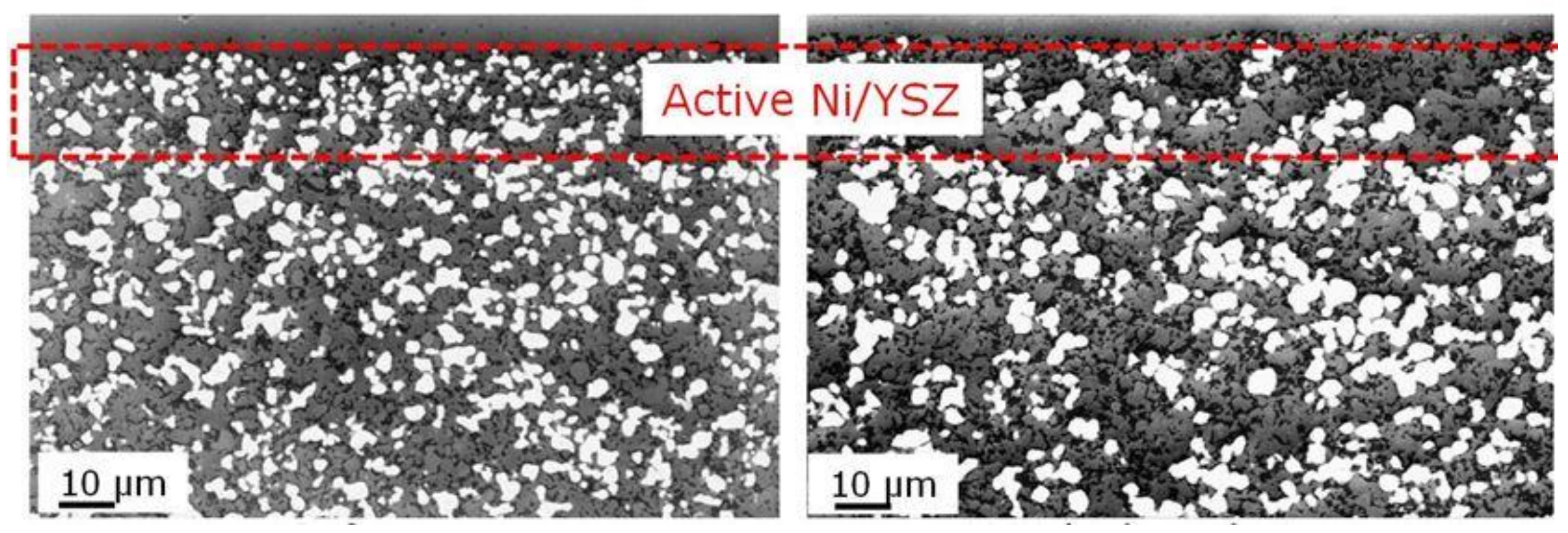
Results and discussion



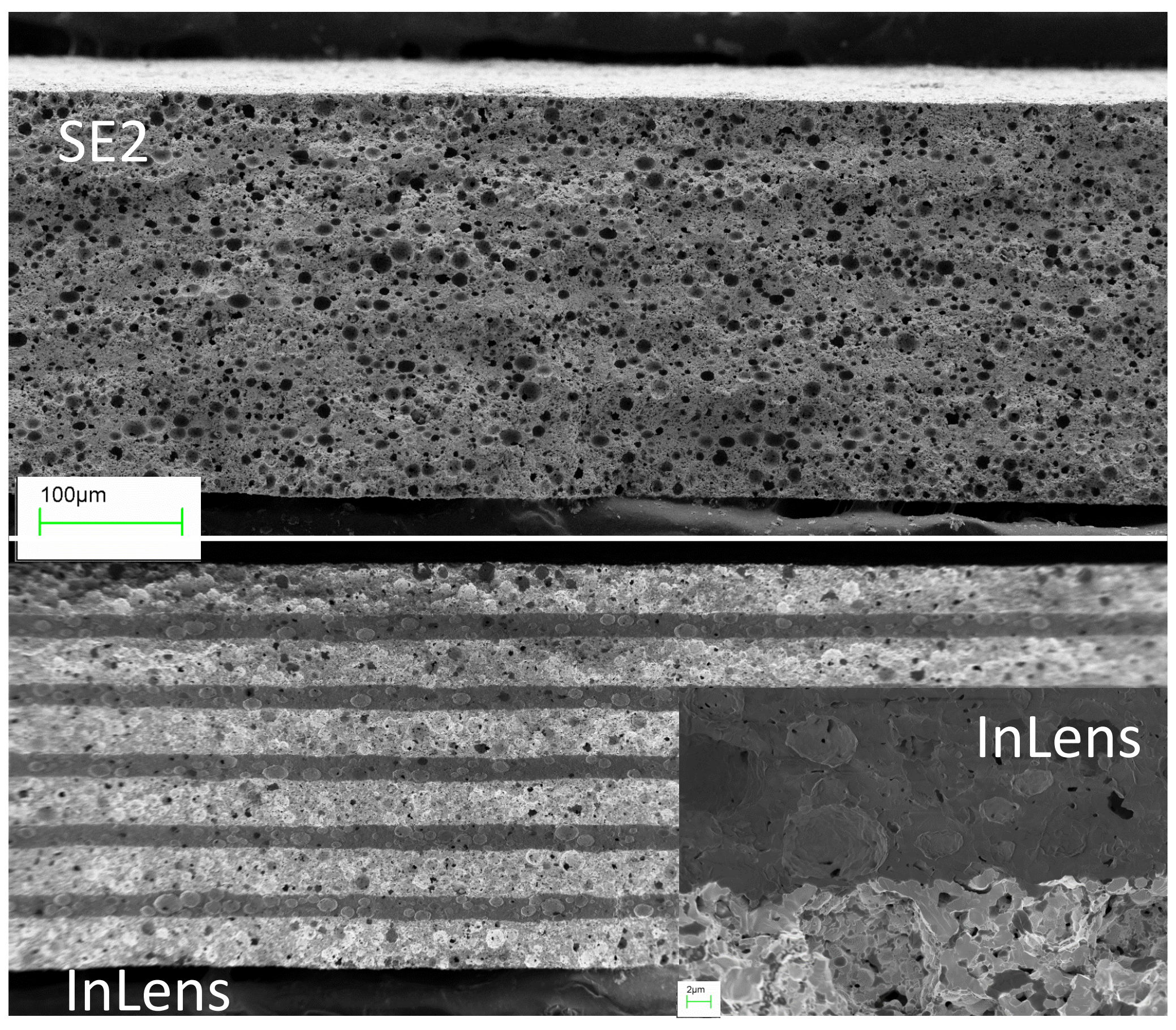
Principle of a solid oxide electrolyzer cell (SOEC), and illustration of the tracking of the electronic pathways in both electrodes .



SE2 and InLens images revealing the electronic pathways.



Visualization of the degradation of the contact between the Ni/YSZ electrode and the YSZ electrolyte in a solid oxide electrolyzer cell. Left image: Reference. Right image: After more than 4000 h of electrolysis.



Visualization of electronically percolating phases in electrodes for devices consisting of LSCM/CGO for electrochemical reduction of NOx from exhaust gas from vehicles.

References

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2. D.C. Joy, Compiled experimental electron scattering data http://www.mc-set.com/science/eesd/lb_load.php

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