

## Developing a high-temperature solid state electrochemical lab in the TEM

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### Background incl. aims

Conducting both electrochemical and structural/compositional analysis simultaneously and on a nanoscale level enables a direct correlation between electrochemical performance and the material's properties, tracking their changes over time and different operational conditions<sup>1</sup>. The aim of the presented work is to establish a solid method that combines high-temperature solid state electrochemical analysis with (S)TEM<sup>2,3</sup>. Such a method is crucial for advancing solid oxide electrolysis and fuel cells (SOEC/SOFC) and can be relevant to other solid-state electrochemical technologies such as solid-state batteries and thermoelectric devices.

### Methods

A method development is presented in which MEMS chip-based heating-biasing TEM holders are combined with environmental TEM (ETEM) and a potentiostat for electrochemical impedance spectroscopy (EIS). Various preparation methods and TEM sample geometries are included, such as model solid oxide cells prepared by pulsed laser deposition (PLD), along with an advanced FIB-SEM sample preparation procedure. Operando high-temperature electrochemical (S)TEM experiments are conducted.

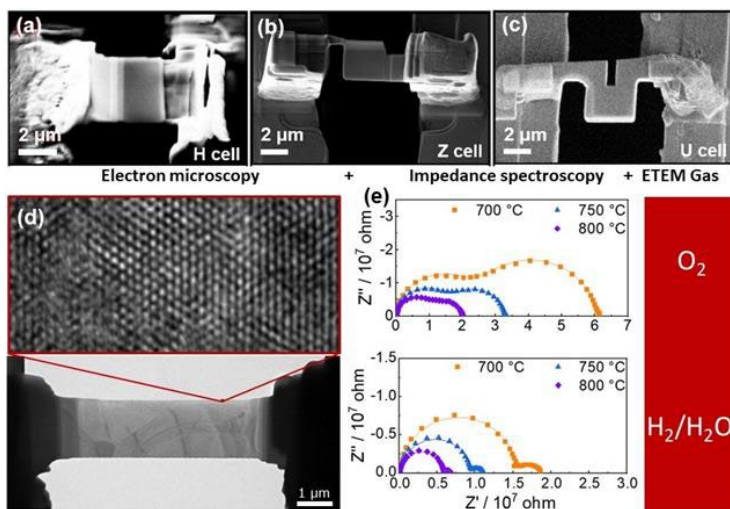
### Results

The feasibility of conducting electrochemical cell tests in the (S)TEM is demonstrated, including reliable EIS measurements of full solid oxide cells at temperatures up to 800 °C in gases such as O<sub>2</sub> and H<sub>2</sub>/H<sub>2</sub>O and with applied electrical polarization<sup>1,2</sup>. The presentation will discuss challenges and necessary requirements for performing such experiments<sup>2</sup>. Examples of EIS analysis in the TEM will be presented<sup>1,2</sup>. Additionally, results from experiments are presented where polarization-induced degradation at an electrode-electrolyte interface in an SOEC was indicated by EIS analysis and confirmed by electrochemical operando STEM observation.

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**Graphic:**



**Keywords:**

Operando (S)TEM, electrochemistry, SOEC, SOFC

**Reference:**

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3. S.B. Simonsen, et al., *Microsc. Res. Tech.* 86 (2023) 1003-1011