Research Project #8

Designing SOFC Electrodes via Correlative *In-Situ* TEM and 3D Atom Probe Tomography (IET-1 & ER-C-2)

The solid oxide fuel cell (SOFC), which promises the highest efficiency of all fuel cell types, and synergistically related solid oxide (co)electrolysis cell (SOEC) are thought to be the pillars for paving the path towards net carbon zero society. Currently though, the durability of electrodes during long-term operation limits the cycle life of these cells. At the Institute of Energy Technologies - Fundamental Electrochemistry (IET-1), we are working with this topic for many years and have developed industrially promising ceramic-based electrode materials for this purpose **[1,2]**. However, further material engineering is necessary to ensure long-term catalyst activity before these electrodes can be integrated into a commercial system.

Building upon the success of the recent HiTEC project (2022-2025) that established our *in-situ* TEM framework and led to new insights into 2D catalyst exsolution dynamics **[3,4]**, this project will take the crucial next step. This project for the first time would combine two state-of-the-art non-trivial techniques for the SOFC materials, creating a unique, closed-loop feedback system for materials development. Utilizing the newly developed *in-situ* gas-heating-biasing workflow at relevant atmospheric pressure, we will be directly observing catalyst exsolution, activation, and degradation during electrochemical processes. Following the *in-situ* experiment, we will then transfer this same sample to the new TEM-APT (TOMO) **[5]** microscope at Ernst Ruska Centre for Microscopy and Electron Spectroscopy - Materials Science and Engineering (ER-C-2) to conduct a 3D atomic-scale dissection. This will provide the first-ever 3D atomic maps of element distribution at the exsolved catalyst/ matrix interfaces after they have been observed functioning in real-time. These insights will provide the "ground truth" feed back to our materials synthesis group for synthesis of next-generation, degradation-resistant materials.

This project is the ideal bridge between the materials-science mission of IET-1 and the world-class microscopy capabilities of ER-C-2. As the lead institute and HITEC member, IET-1 will be the hub for *in-situ* science, electrochemistry, and materials synthesis. Its responsibilities will include: leading the project and defining the key scientific questions; leading the *in-situ* (gas-heating-biasing) TEM experiments and the FIB-SEM sample preparation; working with its internal “Solid-State Electrolysis & Fuel Cells” focus group to perform all macro-scale electrochemical testing (button cells, EIS) to validate device-level performance; to design and driving the final nano-to-macro correlation and produce new generations of exsolving perovskites. Our cooperation partner, ER-C-2, provides the unique, indispensable infrastructure and expertise for the final atomic-scale analysis. It will be responsible for: providing expert consultation for the APT-specific sample preparation workflow, ensuring high-quality data from the TOMO microscope; executing the novel correlative TEM-APT (TOMO) experiments on the *in-situ* TEM samples; leading the 3D atomic data reconstruction and analysis from the APT; and providing access to and support for its world-class microscopy infrastructure.

For this purpose, the following tasks will be addressed during the three years tenure of the Ph.D.

WP1: Lead *in-situ* gas-heat-biasing TEM experiments - test the degradation hypothesis by tracking dynamic changes in electrode activation, catalyst exsolution, and degradation under electrochemical load.

WP2: Develop and execute a robust FIB-SEM workflow to prepare APT needles from the *in-situ* TEM samples. Execute transfer of the *post-in-situ* sample to the TOMO microscope for correlative 3D Atom Probe Tomography.

WP3: Perform TEM and APT investigation from relevant macro-scale samples. Correlate *in-situ* and macro-scale results to establish a quantitative structure-property-degradation relationship, creating a "materials feedback loop" to inform the design of new materials.

Thus, while working in a highly multidisciplinary, multicultural spirited team at the end of the Ph.D., the candidate will not only master the specific skillsets required to operate advanced TEMs, or design *in-situ* TEM experiments, but also gain in-depth knowledge of industrially relevant SOFC/SOEC processes.

Location of the HITEC Fellow	Forschungszentrum Jülich, Institute of Energy Technologies - Fundamental Electrochemistry (IET-1), Director: Prof. Dr. Rüdiger- A. Eichel https://www.fz-juelich.de/en/iet/iet-1
Partners of the HITEC Project	Forschungszentrum Jülich, Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons - Materials Science and Technology (ER-C-2), Director: Prof. Dr. Joachim Mayer https://www.fz-juelich.de/en/er-c/er-c-2
Specific **requirements**	M.Sc. degree in Materials Science/Physics/Chemistry/ Mechanical Engineering; in-depth understanding of material characterization
For **project specific** questions please **contact**	Dr. Shibabrata Basak, IET-1, s.basak@fz-juelich.de

[1] F. Kunz et al., ECS Transactions, 2023, 111 (6),1667, DOI 10.1149/11106.1667ecst

[2] S.E. Wolf et al., Electrochemistry Communications, 2024, 16, 107799, DOI: 10.1016/j.elecom.2024.107799

[3] P.K. Chakraborty, Nano Today, 2025, 61, 102649, DOI: 10.1016/j.nantod.2025.102649

[4] S.E. Wolf et al., Journal of Materials Chemistry A, 2025, 13, 34565-34584, DOI: 10.1039/D5TA04819B

[5] A. Vayyala et al., Microscopy and Microanalysis, 2024, DOI: 10.1093/mam/ozae044.039