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In Situ Probing of transition metal-oxide heteroInterfaces for high-peRformance solid-state Energy devices

Reporting

Project Information

INSPIRE

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[Project website](#)

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Periodic Reporting for period 1 - INSPIRE (In Situ Probing of transition metal-oxide heteroInterfaces for high-peRformance solid-state Energy devices)

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[Summary of the context and overall objectives of the project](#)



The current solid oxide cell (SOC) technology has been optimized to operate at high temperatures (> 750 °C) but suffers from high maintenance costs and microstructural and chemical degradation issues. These problems can be expected to decrease by operating at lower temperatures (300-500 °C). However, today's state-of-the-art materials and microstructural designs lead to an overall performance decrease at lower temperatures due to slower ionic/electronic motion. In recent years, advances in material and microstructural designs have presented unparalleled opportunities for further development. For example, through careful tuning of the crystal structure and the stress/strain, thin films exhibit orders of magnitude faster ion-exchange/diffusion kinetics. However, there is a continuous debate regarding the origin of these enhancements, mainly due to the lack of systematic and comparative analysis with surface-sensitive in situ and ex situ characterisation techniques. INSPIRE addresses this issue by a combination of spectroscopic techniques; low energy ion scattering spectroscopy, X-ray photoelectron spectroscopy and secondary ion mass spectrometry. In terms of materials design, we tune the crystal structure in two ways: the first is by varying the substrate temperature during pulsed laser deposition (PLD) and the second is by depositing vertically aligned composite nanostructures (VAN) by PLD. The performance of these heterostructures is investigated for LT-SOC applications, targeting higher performance outputs and stability at lower operating temperatures (300-500 °C).

INSPIRE aimed to bring together fundamental knowledge and materials engineering by combining advanced elemental characterization techniques with next-generation film microstructures. One of the key metrics of this project was to use materials based on our existing knowledge in electrochemistry and nanotechnology. In this way, our engineered microstructural designs will provide researchers with new directions to explore, while providing commercial organizations with valuable information to adapt these microstructures with familiar materials for low-temperature operation.

The overall objective of this project is to probe the remarkable activity in the thin film electrodes (single-phase or vertically aligned nanocomposite) with the latest instrumental capabilities. A deeper understanding of the underlying kinetic mechanism helps to rationally design other heterostructures and heterointerfaces with superior properties, targeting durable, high-performance, and low-temperature operations. The second objective is to monitor the electrochemical performance for LT-SOC applications systematically. The study on VAN heterostructures as well as their single-phase counterparts reveals the influence of strain and lattice interactions on the performance of these cathodes.

Work performed from the beginning of the project to the end of the period covered by the report and main results achieved so far

The project initially evaluated a class of single-phase lanthanum strontium cobaltite $\text{La}_{1-x}\text{Sr}_x\text{CoO}_{3-d}$ (LSC) materials, $\text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-d}$ (LSC64) and $\text{La}_{0.8}\text{Sr}_{0.2}\text{CoO}_{3-d}$ (LSC82), as air electrodes for SOCs. The crystallinity of the films was studied as a function of substrate temperature during pulsed laser deposition and post-thermal annealing. Three crystalline states, i.e. fully crystalline, partly crystalline, and amorphous films, were chosen for further studies. Chemical activities and elemental

distribution of the films subjected to long-term stability tests and the as-prepared films were compared. Our investigations on LSC64 films revealed that less crystalline films were not catalytically active for low-temperature (400 °C) SOC operation. At intermediate temperatures (500 and 600 °C), a comparable activity was measured for both fully crystalline and partly crystalline films. However, the degradation rate of the partly crystalline film was two times slower than the fully crystalline one for 100 h of testing. Based on all the findings, we determined that initial microstructural features of the films such as grain size, grain orientation and topography determine the cation segregation behaviour and thus the long-term catalytic activity.

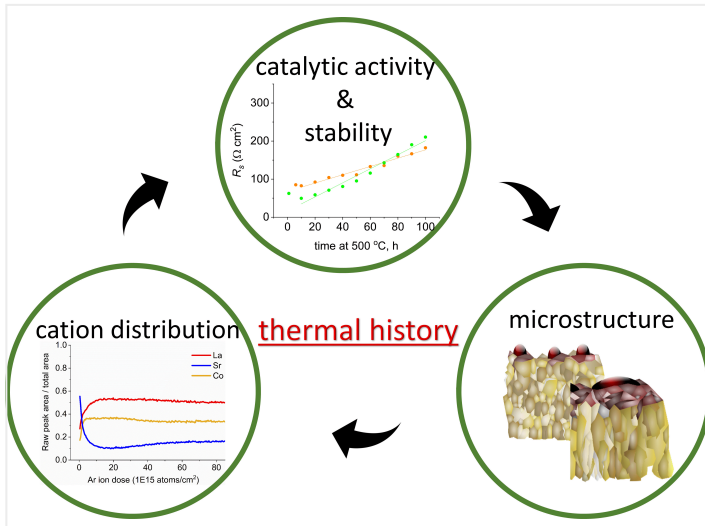
In the second part of the project, vertically aligned composite nanostructures (VAN) comprising two materials known to show exceptionally fast electrokinetics for low temperatures operations were selected. This was a combination of an electronically conducting Ag metal and an ionically conducting oxide $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_2$ (GDC). The Ag-GDC VAN films were deposited epitaxially on oriented yttria-stabilized zirconia substrates by PLD. Preliminary results show successful growth of ~40 to 400 nm thick oriented Ag/GDC VAN films. Both phases grew homogeneously and epitaxially on the substrate. *in situ* Raman spectroscopy was used to probe the stability of the films under airflow from room temperature to 400 °C. Initial findings indicate resistance to oxidation of Ag up to 400 °C and microstructural integrity up to ~350 °C. Additionally, we were able to show that the addition of GDC stabilized Ag by surrounding it through vertically grown columns.

For the exploitation and dissemination of the project, various activities such as publishing research articles, scientific talks at conferences and meetings, and participation in public engagement events were carried out. In terms of publishing, 2 peer-reviewed articles and 2 proceedings papers were published. At least 2 more peer-reviewed articles linked to INSPIRE and an invited chapter contribution to an e-book on nanoengineering methods developed and utilized for SOC air electrodes will be produced. In terms of scientific engagements, oral and poster presentations were given in the following conferences: Solid State Ionics (SSI), PyeongChang Korea in June 2019, European SOFC and SOEC Forum (EFCF) (virtual talk) in October 2020 and Electrochemical Society (ECS Prime) (virtual talk) in October 2020. Additionally, invited talks were given in the UK Surface Analysis Forum (UKSAF) in London, UK in January 2020, and STFC Batteries Early Career Researcher Conference in Oxford, UK in March 2019. In terms of public engagements, the principal investigator participated in the European Researcher's Night in London, UK in September 2019.

Progress beyond the state of the art and expected potential impact (including the socio-economic impact and the wider societal implications of the project so far)

The findings of this project significantly filled the knowledge gap regarding the performance and durability of SOC devices. The results were widely disseminated via oral and written communications. The project allowed the principal investigator (O. Celikbilek) to start her independent research career. Within the duration of the project, she has received training on cutting-edge surface characterisation instruments, attended personal and professional development workshops, trained, and mentored several Master and PhD students. The experience and knowledge gained in this project helped Dr

Celikbilek to find her new role as CNRS postdoctoral researcher in France, leading the new EU FET-Proactive project, EPISTORE, in one of the partner institutes (LMGP, CNRS) on the development of thin-film Reversible Solid Oxide Cells (TF-rSOCs). EPISTORE's aims are in perfect alignment with INSPIRE and therefore will provide Dr Celikbilek with further opportunities to advance in the subject and to continue contributing toward research and development within the EU. The encouraging outcomes of the INSPIRE programme will lead to further investigations on prototype devices and may necessitate an application for IP protection in the future.



thermal history effects on catalytic activity and stability of thin films

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