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Operando optical techniques for understanding materials and interfaces in energy applications

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Ion-based devices such as batteries, fuel cells and electrolyzers will play a major role in the future carbon-free energy systems. Many efforts are being dedicated to find techniques that allow understanding interfacial and ion diffusion phenomena occurring during operation, as these are often limiting overall performance. All the developed characterization technique show different compromises between spatial and time resolutions, sensitivity, level of detection etc., which can be solved by a combination of different approaches.
We have developed procedures based on optical techniques: Spectroscopic Ellipsometry (SE), and tip-enhanced Raman spectroscopy (TERS). Despite the well-known capabilities of SE to infer the properties of thin film and multilayers, such as thickness, crystallinity, materials ratio in mixtures, roughness, structure of the interfaces, electronic band structure etc., the use of this affordable, non-destructive technique for the study of ion-transport under operation is very so-far limited. We will illustrate the potential of SE with several examples on the field of lithium-ion batteries and solid oxide cells. In a first example, we will show the use of SE to monitor Li+ transport properties and degradation phenomena through real-time tracking of the oxidation-state and volume changes associated with lithium insertion and extraction along LiMn2O4 and Li4Ti5O12 thin-film electrodes in different liquid electrolytes (LiSO4 and LiPF6, respectively). In a second example, we use SE for studying the concentration of point defects in La1-xSrxFeO3-δ (LSF) thin films as a function of equivalent oxygen partial pressure, temperature and Sr concentration . Finally, we present our recent advances in the utilization of Tip-Enhanced Raman Spectroscopy (TERS) for the study of the distribution of species in the surface of Li-ion battery cathodes. We show the high potential of TERS for studying phase evolution at grain boundaries thanks to the combination of the chemical sensitivity of Raman
The development of the operando techniques is carried out in the frame of different projects, which also provide relevant samples from the state-of-the art of Li-ion batteries and SOFC. The most important are: i) EPISTORE (FETPROACT-EIC-07-2020), ii) 3D-ASSET (TED2021-129663B-C51), iii) AfreeSSB (M-ERA.NET PCI2022-132960), iv) ADVAGEN (Horizon Europe 101069743), and v) HARVESTORE (H2020-FETPROACT-2018-2020). The optical procedures applied to the model system arising from those projects, combined with synchrotron operando techniques would provide a deep insight in the mechanism limiting the performance and durability of materials and devices.

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