In-Situ Characterization of Thin Films with Raman and X-ray Absorption Spectroscopy

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Thin films of metal oxides and transition metal oxides are nowadays an established asset in a variety of sectors due to their unique features and variety of uses, including energy storage, catalysis, and optoelectronics. Understanding the structural and chemical properties of thin films is essential in order to develop new functional materials. Raman and X-ray absorption spectroscopy (XAS) are among the most effective non-destructive technique that are currently used to study this kind of materials. They can provide complementary informations about the atomic and electronic structure, the presence of defects and the overall structural order of the sample. Combined with adequate *in-situ* setup, these techniques enable real-time monitoring of structural changes and chemical reactions occurring in the bulk as well as in the superficial region of the sample [1].

In this seminar, we will describe two novel *in-situ* cell for Raman and X-ray absorption spectroscopy. With the Raman cell, the structural changes of transition metal oxides can be studied as a function of the temperature and atmosphere. As a case study, we will discuss about the redox process occurring within MoO_3 thin films as a function of temperature and air partial pressure [2].

On the other hand, the XAS cell is an electrochemical cell modified to study the structural dynamics of an electrode material in *operando* conditions. It allows to acquire XAS data in fluorescence mode while flowing a thin electrolyte layer above the sample while applying a voltage. As a case study, we will discuss about thin films of Ni-doped maghemite on Pt substrates, tested as a possible catalyst for the oxygen evolution reaction (OER). Via XANES and EXAFS analysis, we will discuss about the local environment of the Ni dopant as a function of the Ni/Fe ratios and how the resulting structure change the catalyst performances for OER [3].

[1] F. Paparoni, E. Mijit, S. Kazim, M. Minicucci, N. Pinto, A. D'Elia, S. Macis, C. Kim, S. Huh, R. Gunnella, A. Marcelli, A. DiCicco, and S. J. Rezvani, *Metallic Interface Induced Ionic Redistribution within Amorphous MoO_3 Films.*, *Adv. Mater. Interfaces* **2022**, *9, 2200453.*
[2] F. Paparoni et al., *Raman Cell for In Situ studies at High Temperature in Controlled Ambient*, (in progress).
[3] F. Paparoni et al., *A novel electrochemical flow-cell for operando XAS investigations on x-ray opaque supports* (in progress).