In-situ Electrochemistry at the Nanoscale in an Electron Microscope

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The PhD topic aims at developing an original methodology for the in situ investigation of electrochemical processes and phenomena induced by the application of an electric field, in nano-materials at the nanometre scale. The approach is based on the use of transmission electron microscopy (TEM) carried out on nanomaterials placed in a sealed environmental liquid cell equipped with a three electrodes set-up [1]. This innovative technology has never been applied to energy conversion devices, especially hydrogen production (hydrogen evolution reaction - HER and oxygen evolution reaction - OER) through electrolytic water splitting, and for the oxygen reduction reaction (ORR) in fuel cells. As a first approach, we will quantitatively explore the behaviour of specific systems of high interest in electrocatalysis: nanoscaled mixed oxides, metal phosphides and borides [2, 3]. Materials stability, morphological, structural, chemical, electronic changes and reaction mechanisms will be investigated during electrochemical processes, especially hydrogen and oxygen evolutions. The project will take advantage of state-of-the-art synthesis protocols for providing a range of original nanoparticles active for the OER, ORR or HER. The development of new in situ characterization tools is a requirement to understand and enhance the performances and aging behavior during real-life use of energy storage and conversion devices. Up to now, only very little data is available about the behaviour of the electrocatalysts in water under the application of an electrical potential. This informations are important for the understanding of the electrochemical reactions and stability of the electrocatalyst.

IPCMS and LCMCP teams have developed during the last years a strong collaboration on advanced characterization of novel nanomaterials. This collaboration has been recently extended to in situ environmental TEM emphasizing gas-assisted chemical reactions addressing fundamental topics in materials science. The present PhD topic will generate in France a new methodology for in situ direct monitoring of materials for electrocatalysis and thus provide unprecedented insights on the dynamics of complex systems employed in this field. The activity of the IPCMS - TEM group focuses on the study of the structure, chemical composition and organization of nanomaterials. The development of in situ methodologies has become one of the group's priorities. The "Hybrid Materials and Nanomaterials team from the LCMCP gathers experts in the synthesis and characterization of oxides, hybrid and non-oxide materials and nanomaterials. The team is proficient in the study of electrochemical properties of materials dedicated to energy conversion, water splitting and metal-air batteries. The compounds can be adequately processed to apply more traditional electrochemical characterization tehcniques available at LCMCP. The PhD student will participate in setting-up the experimental in-situ TEM device and carry out experiments by using the eletrochemistry cell at IPCMS, and he will be closely involved also in the materials synthesis and characterization at LCMCP.

[1] M. E. Holtz, D. A. Muller et al, **NanoLetters 14**, 1453, 2014.
[2] H. Vrubel, X. Hu, **Angew. Chemie Int. Ed. 51**, 12703, 2012.
[3] Schaak et al, **J. Am. Chem. Soc. 135** (25), 9267; 2013.