MS14-01 **Operando characterization of lithium battery electrode materials.** Guy Ouvrard,[a] Miloud Zerrouki,[a] Christian Masquelier,[b], Mathieu Morcrette,[b] Stéphane Hamelet,[b] and Stéphanie Belin, [c] [a] *Institut des Matériaux Jean Rouxel,Université de Nantes -CNRS, Nantes, France,* [b] *Laboratoire de Réactivité et de Chimie des Solides, CNRS - Université de Picardie Jules Verne, Amiens, France,* [c] *Synchrotron SOLEIL, Gif s/ Yvette, France*
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Lithium battery materials evidence redox processes and structural changes, directly related to the electrochemical performances: energy, capacity, reversibility and life time. This is due to the reversible insertion of lithium ions and electrons. Many characterization techniques are used in order to explain the general behaviour of these materials. Most of them are only operating after equilibrium of the battery, and even after withdrawal of the material under study. Nevertheless, in order to improve positive electrode performances, it is of primary importance to deeply characterize these materials during the battery cycling, beyond the electrochemical characterization. Among the very few techniques available for *operando* studies, X-Ray Diffraction (XRD) and X-ray Absorption Spectroscopy (XAS) appear very powerful, with the high spatial and time resolutions provided by synchrotron radiation. Moreover XAS is well known for its capabilities in the charge transfer and structure determinations, both being the major changes induced by cycling of electrode materials. By an appropriate combination of three XAS beam lines and one XRD beam line, in using a specially designed electrochemical cell [1], we have studied positive electrodes made with $LiFePO_4$ as the active material. In a first step, XRD experiments have confirmed the two-phase process of the lithium insertion/deinsertion and observed a possible delay in the phase transformation. Subsequent experiments by XAS put in evidence a significant heterogeneity in the behaviour of the electrode in the operando mode. We put in evidence the heterogeneity of the electrode during the functioning, some parts being delayed and others advanced, as compared to the mean charge state of the electrode. A mapping of this heterogeneity was made at different scales. Such heterogeneities depend on various parameters: cycling rate, particle size, pressure, electrode formulation and preparation.

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MS14-02 **An eye on the inside: imaging of catalytic particles under reaction conditions** Andrew M. Beale[1], Simon D. M. Jacques[2], Matthew G. O'Brien[1], Marco Di Michiel[3] and Bert M. Weckhuysen[1] [1]*Inorganic Chemistry and Catalysis, Utrecht University, The Netherlands,* [2]*School of Materials, Manchester University, UK*, [3]*ID15, ESRF, France*
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Industrial catalysis utilizes mainly μm to mm-sized catalyst particles in catalytic reactors instead of powders since this minimizes problems associated with for example back pressure and clogging. In recent times, efforts have been made to study and characterize these 'real life' single particles so as to determine the nature of chemical species present in 2D and 3D during various stages of the catalyst lifetime such as preparation, reaction and deactivation. Traditionally this sort of analysis is performed on ex situ samples using invasive approaches which often interfere with the chemical process under study and the subsequent conclusions that can be drawn. As a result there has been a recent move towards studying these processes non-invasively and where possible, dynamically in order to understand in detail how the chemistry evolves within catalyst particles and how this and the spatial distribution of the various chemical components influence catalytic behaviour. For this purpose we have developed the technique of synchrotron-based X-ray Diffraction Computed Tomography (XRD-CT) for imaging catalytic solids in real time in order to examine how the active phases form, how they behave under reaction conditions and why they eventually deactivate [1,2].

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