Analysing Mesoporous Thin Films Electrodes by Glancing-Incidence XRD - Application to Photorechargeable Li-Ion Batteries

Olivier Nguyen¹, Natacha Krins¹, Gianguido Baldinozzi², Christel Laberty-Robert¹

¹Laboratoire de Chimie de la Matière Condensée de Paris, UPMC / Collège de France, CNRS UMR-7574, Sorbonne Universités, 4 Place Jussieu, 75005 Paris, France
²SPMS, LRC Carmen, CNRS CEA CentraleSupélec, Université Paris-Saclay, Gif-Sur-Yvette, France

Outline

Photo-rechargeable lithium-ion batteries, which are able to both harvest and store solar energy within electrodes, are a promising technology for a more efficient use of intermittent solar radiation [1]. However, there is a lack of understanding on how the light-induced lithium extraction reaction occurs within the electrode host lattice. In fact, this technology is based on bifunctional nanostructured electrodes, which undergo simultaneous complex modifications: structural reorganization through lithium insertion and photo-excited charges creation through solar illumination. In order to get more insight on the mechanism governing the photo-recharge of a single device based on a TiO₂ thin film electrode made of mesoporous anatase, we perform ex-situ glancing-incidence XRD experiments. Using laboratory glancing incidence diffraction, and implementing proper corrections for the instrumental broadening of this diffraction geometry in the Rietveld refinement software XND, it was possible to extract quantitative information about the structure (lattice, strain, and atomic positions) and the microstructure (crystallite size and micro-strain), selectively probing the material on a depth of few nanometers.

Glancing Incidence XRD [2]

Snell, Fresnel and XRD

Polycrystalline Thin Film

Angle of incidence \( \alpha \)

Diffraction angle, \( 2\theta \)

Sample rotation, \( \psi \)

Sample inclination, \( \nu \)

Analyzer Crystal

Goebel mirror

Tunable Electrode Architecture

1 layer – 100nm

5 layers – 500nm

14 layers – 1500nm

SEM

FEG

Mesoporous TiO₂ Anatase thin film dipped on FTO/glass

Conclusion

- The characteristics of architected mesoporous TiO₂ thin films, produced by dip-coating on a FTO/glass substrate, can be studied across their thickness.
- Probing the films at multiple glancing angles shows evidence of single-phase nanocrystalline anatase with a same structure and microstructure across the entire film thickness, a result in very good correlation with SEM-FEG and TEM analyses that involve the destruction of the sample.

Thin Film Electrode Synthesis

References

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Outline

Probing

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