High Resolution Analytical Tools for Industrial Applications

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The enhanced industrial applications of new nanostructured materials, as well as the need to understand the behaviour, as well as the degradation processes, of older materials with structure at the nanometer level, necessitate an increased use of High Resolution Transmission Electron Microscopy for structure analysis and, even more, for analytical analysis at the sub-nanometer level.

For these reasons, the Hydro-Quebec Research Institute has recently acquired two newly designed high-end microscopy tools. The first one is a new Focused Ion Beams (NB5000 NanoDUE'T) with focused ions and electron beam system from Hitachi. This FIB-SEM has a vertical ion beam for higher machining precision, an accelerating voltage that can go up to 40 kV and a current that can be as high as 60 nA, for rapid sample throughput. This system is equipped with a high resolution SEM, having a Shottcky emitter with a resolution below 1.0 nm at 15 kV, and with a Bright Field STEM detector that has a resolution below 0.8 nm at 30 kV. It is equipped with an SDD EDS detector and will shortly be equipped with an EBSD camera. This tool is ideal to rapidly prepare samples for HRTEM observations but also to perform high resolution analysis by itself. Also combined with a 3D sample holder, with 360o rotation, it is possible to prepare samples for rapid HRTEM tomography analysis. Figure-1 presents such a 3D pin sample of Zr-2.5Nb alloy used in CANDU reactors as pressure tube materials.

The second system is a Hitachi HD2700C dedicated STEM with a CEOS aberration corrector and a cold field emission gun, equipped with an EELS Spectrometer (specially designed Enfina from Gatan) and a newly designed SDD EDS detector from Bruker. The microscope was installed in an isolated refurbished building, inside a commercially available cold storage room with magnetic shielding and equipped with a temperature control system that limits temperature fluctuations to about 0.15 oC in a 24 hours period. Less than two weeks after the beginning the installation, an information resolution of 78 pm was achieved in HAADF (FFT analysis on a 110 Si sample).

The major goal of using these two systems is to understand rapidly the relation between the nanostructure and the macroscopic properties of new industrial materials, in such fields as new batteries materials (Figure-2) or new catalysts but also in more common industrial products such as electroformed iron (Figure-3) or steel. This combination of tools offers the possibilities to investigate rapidly the structure and composition in 3-dimensions of very complex materials. A number of examples will also be presented [1].

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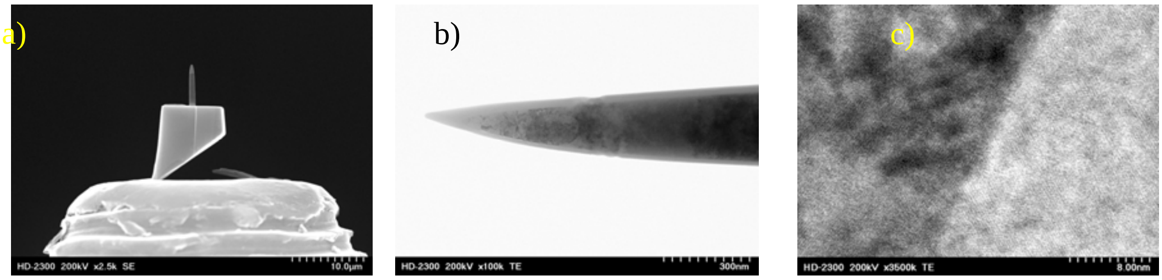

Fig.1 a) Low magnification SEM micrograph of a FIB tomography sample of a Zr-2.5Nb sample, with Bright-Field STEM images at **b)** low-resolution and **c)** high-resolution, revealing easily the complex structure of this sample (these three micrographs were obtained on a HD-2300 dedicated STEM at 200 kV).

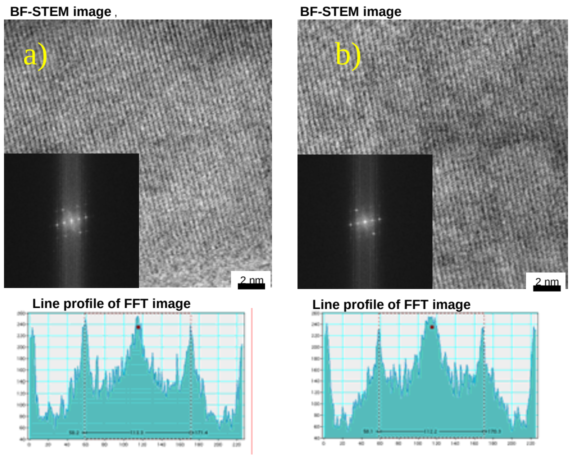

Fig. 2 Bright-Field High-resolution FEG-STEM of a $LiFePO_4$ crystallite after partial discharging, a) from the border of the crystallite and b) from the center. Variation in spacing can be related to the changed in lithium content.

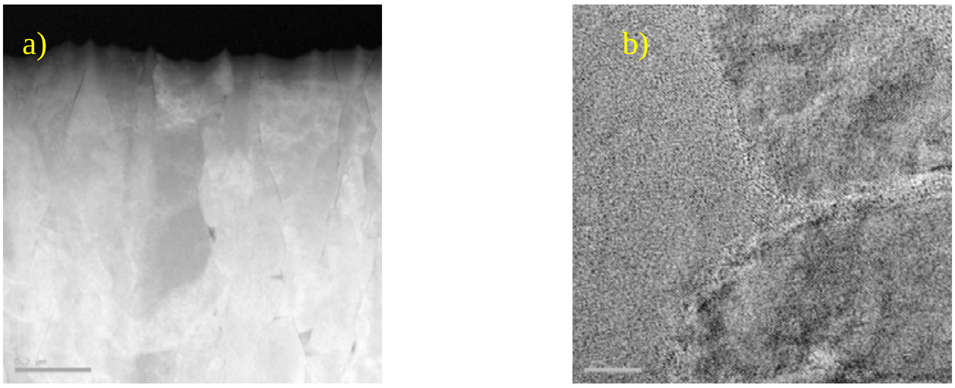

Fig. 3 a) DF-STEM image of electroformed iron sheets showing the presence of low elements at grain boundaries and **b)** high resolution BF-STEM images showing a triple junction in this materials. EDS analysis of the grain boundaries indicate the presence of O, S, Cl and C, coming from the electrodeposition process.

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