

On the better understanding of Li/polymer interfaces by means of live Scanning Electron Microscope Observations Coupled with AC-Impedance

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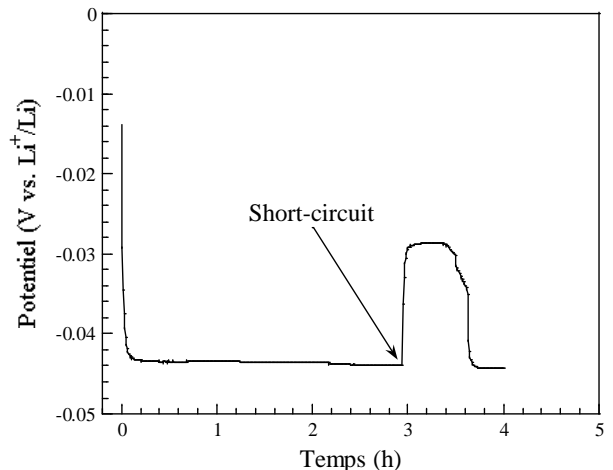
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Rechargeable batteries are the key components of today's portable electronics involving cellular phones, portable computers, and others. However, which of the battery technologies considered, its autonomy and more specifically its lifetime mainly depend on the control of the electrode/electrolyte interface upon cycling. This is more so for Li rechargeable batteries whose use, despite their attractive performances, has been plagued by the uncontrolled dendritic growth at the Li surface upon cycling. To get a better insight in these critical interface issues, recent efforts have been devoted towards developing new ways to perform in situ measurements. Recent advances in instrumentation have boosted the implementation of in situ Scanning Electronic Microscopy (SEM) techniques to the field of energy storage [1]. Mid-term towards our goals, we reported the feasibility, by means of a specific transfer system designed by Philips, to perform semi in situ electron microscopy measurements enabling post-mortem investigation or interface characterization without ever exposing the sample to air. Through such a study we confirmed that Li deposit morphologies are strongly dependent on current density with mossy lithium at low current and dendrites at high current.

Pursuing further our efforts we recently succeeded [2] in modifying our system so that we can now routinely perform in situ scanning electron microscopy while cycling the battery within the microscope. For instance, we demonstrate the feasibility of cycling a Li/Polymer/LiV₃O₈ cell within the microscope for more than five cycles while performing AC impedance and collecting SEM shots. However, among the most spectacular results are 1) the live growth of dendritic Li as function of time on the copper electrode of a Cu/Li based polymer/Li cell that was constantly polarized, and 2) the first ever reported direct visualization of the fusible effect caused by a dendrite, which translates by a rectangular wave anomaly in the voltage variation as a function of time for a polarized Li/polymer/Li cell (Fig. 1).

Such SEM studies were complemented by three-electrode impedance measurements that were recently performed in situ within the microscope, and a correlation



was evidenced as will be reported. Through this presentation we will try to convey the message that this new analytical tools are quite powerful in scrutinizing battery interfaces. Further efforts will be placed on duplicating such SEM studies on a plastic Li-ion cell. Due to the electrolyte volatility, we will consider the use of environmental microscopes.

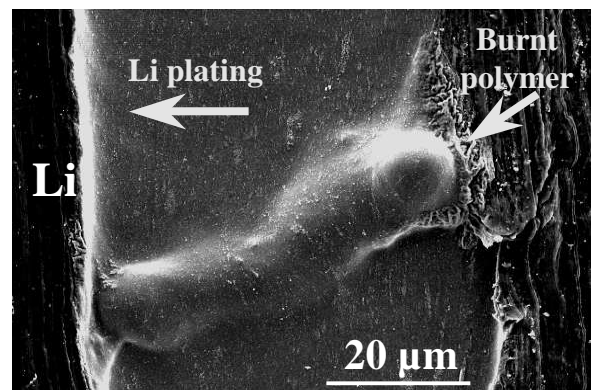


Figure: 1. Voltage profile of a symmetric Li cell placed under polarization (**top**) and cross section of the Li/polymer/Li cell after the shape accident (**bottom**).

References:

- [1] Orsini F., Dupont L., Beaudoin B., Grugeon S., Tarascon J-M., *Int. J. Inorganic Materials*, **2** (2000), 702
- [2] Dollé M., Sannier L., Beaudoin B., Trentin M., Tarascon J-M, *Electrochemical and Solid State Letters* **5** (12) A 286-289 (2002).