Features and Benefits of Pulsed RF GD OES for the characterisation of electrodes of Li-ion batteries

Patrick Chapon, HORIBA Scientific, 16 rue du Canal, 91160 Longjumeau, France

Abstract

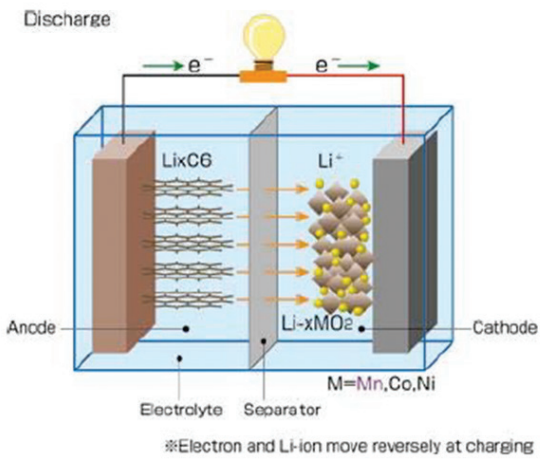
Pulsed RF Glow Discharge Optical Emission Spectrometry provides Ultra Fast Elemental Depth Profiling and is of great interest for the characterisation of both positive and negative electrodes of Li-ion batteries

Key words

Li-ion batteries, positive and negative electrodes, Depth Profile Analysis, gradients, GD OES, Pulsed RF source, Li bell.

Introduction

A Li-ion battery is a rechargeable battery in which lithium ions move between the anode and the cathode creating an electricity flow.



Source Automotive Energy Supply Corporation, 2007

The chemical reactions do not simply take place at the surface of the electrodes but rather affect depths of several tens of micrometers. Pulsed RF Glow Discharge Optical Emission Spectrometry provides Ultra Fast Elemental Depth Profiling of thin and thick films and has been successfully applied for the characterisation of both positive and negative electrodes of Li ion batteries.

HORIBA Scientific has more than 15 references world wide in this field. Most results obtained are protected by Non Disclosure Agreement and cannot be communicated. We therefore invite people interested in the technique to contact us so that we could also describe more in depth, the unique proprietary features of our instrument and set up some performance tests.

Instrumentation

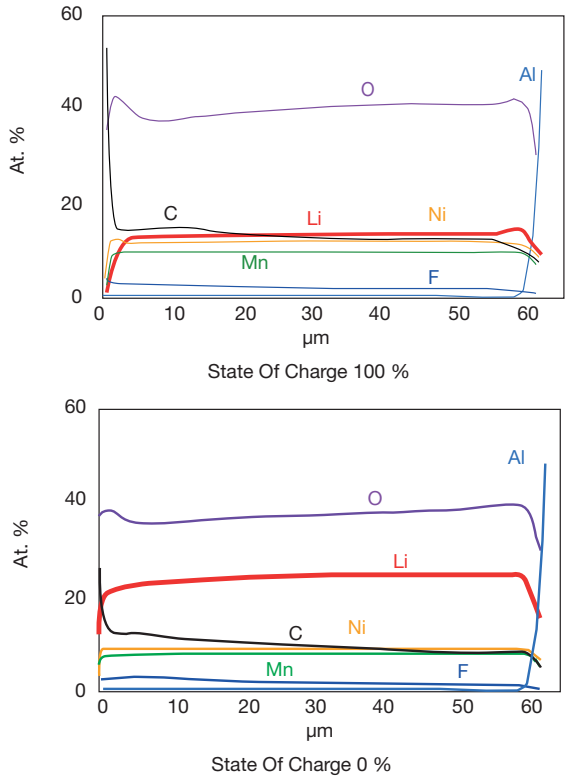
The GD Profiler 2 couples an advanced Pulsed RF Glow Discharge Source to a high resolution, wide spectral range Optical Emission Spectrometer.

The source (GD plasma) permits on one hand a precise and fast sputtering of a representative part (typically 4mm in diameter) of the electrode investigated. Pulsed RF operation is crucial to avoid damaging the fragile electrodes and to prevent unwanted diffusion of the elements during the measurements.

The sputtered species are excited on the other hand by the same GD plasma and the spectrometer simultaneously measures all elements of interest (Li, Co, H, C, Mn, S, O, F, Al, P, Ni, V, Ti, Si, B, Cu etc) as a function of the sputtered depth.

GD Profiler 2

Typical results



Pulsed RF GD OES Depth Profile Analysis of the positive electrode of a battery fully charged and discharged

Key Features

✓Ultra Fast Depth Profiling of both positive and negative electrodes: Typical erosion rate of several microns/minute.

✓ Fast sputtering offers high sample throughput but more importantly a higher sensitivity as more sputtered materials are excited per unit of time.

✓ Ease of Use: The GD source does not require any UHV, the sample to analyse is simply placed against an o'ring facing the anode tube in which the plasma is confined

✓ Simultaneous measurement of all elements of interest

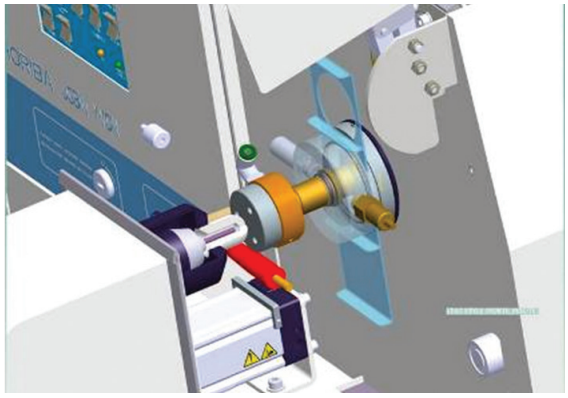
✓ Averaged information over the sputtered area

✓ No thermal effects during measurements with Pulsed RF operation

✓ Provide quick access to embedded interfaces for further SEM observation

Sample handling strategies

The electrodes are often soft and fragile. Successful strategies of sample handling have been developed by HORIBA Scientific to correctly analyse these electrodes including, for the flammable ones, a patented transfer chamber: the "Li bell".



Schematic view of the Li bell mounted on the GD Profiler 2 instrument

Conclusion

Whether you study new electrodes or coatings behaviours, charge and discharge processes, process controls, or perform comparative studies for Li-ion batteries, Pulsed RF Glow Discharge Optical Emission Spectrometry is a valuable companion tool.

References

Investigation of positive electrodes after cycle testing of high-power Li-ion battery cells IV. An approach to the power fading mechanism by depth profile analysis of electrodes using glow discharge optical emission spectroscopy.
Yoshiyasu Saito, Md. Khalilur Rahman. Journal of Power Sources 174 (2007) 877–882

The effects of $LaPO_4$ coating on the electrochemical properties of $Li[Ni0.5Co0.2Mn0.3]O_2$ cathode material.
Han Gab Song, Kyu-Sung Park, Yong Joon Park Solid State Ion. (2012), doi:10.1016/j.ssi.2011.12.014

JOBIN YVON Technology

info.sci@horiba.com
www.horiba.com/scientific

HORIBA Scientific

USA: +1 732 494 8660
UK: +44 (0)20 8204 8142
China: +86 (0)21 6289 6060
France: +33 (0)1 69 74 72 00
Italy: +39 2 5760 3050
Brazil: +55 (0)11 5545 1500
Germany: +49 (0)89 4623 17-0
Japan: +81 (0)3 6206 4721
Other: +33 (0)1 69 74 72 00