#### NANOSTRUCTURED LiFePO4 COMPOSITE ELECTRODES PREPARED by SOL-GEL TEMPLATE SYNTHESIS

# F. Croce<sup>•</sup> \* <sup>§</sup>, C. Sides<sup>^</sup>, B. Scrosati <sup>§</sup> and C. R. Martin<sup>^</sup>

\* Pharmacology Science Department University 'G. D'Annunzio', 66013, Chieti, Italy

### <sup>§</sup> Dipartimento di Chimica, Università di Roma "La Sapienza", 00185, Roma, Italy

#### ^ Department of Chemistry, Center for Research at Bio/Nano Interface, University of Florida, Gainesville, Florida 32611, USA

Rechargeable lithium-ion batteries have reached an established commercial status with a production rate of several millions of units per month. However, this important technological field is still open to improvements. In particular, the present research efforts are focused on the development of new electrode materials in order to increase the energy density content, the power rate, to lower the cost and, at the same time, improve the environmentally compatibility of the battery<sup>1</sup>. Among the several new cathodic materials under development, LiFePO<sub>4</sub> of the phospho-olivine family proposed by Goodenough and co-workers<sup>2</sup> appears particularly interesting due to the low cost and environmental compatibility of its basic constituents.

In this talk we will describe the synthesis, the morphological and the electrochemical characterization of nanostructured Carbon/LiFePO<sub>4</sub> composite cathodes.

A general method for preparing nanomaterials which entails synthesis of the desired material within the pores of a nanoporous membrane has been developed and extensively studied by Martin and co-workers<sup>3</sup>. The membranes used contain cylindrical pores with monodisperse diameters, and a nanoscopic fibril or tubule of the desired material is synthesized within each pores. This method has been used to make tubes and fibrils composed of polymers, metals, semiconductors, carbons, and Li-ion-intercalation materials<sup>4, 5</sup>.

Sol-Gel chemistry is a well known, generally recognized, powerful approach for preparing inorganic materials such as glasses and ceramics<sup>6</sup>. Recently<sup>7</sup>, we have used a patented modification of Sol-Gel method, called CSGP (Complex Sol-Gel Process), to prepare Liintercalation layered oxides<sup>7</sup> of the family  $\text{Li}_x \text{Ni}_y \text{Co}_{1-y} \text{O}_2$  and the iron phospho-olivine compound LiFePO4<sup>1</sup>.

Here we have combined the membrane template method with the CSGP in an innovative Complex Sol-Gel Template Synthesis Process (CSG-TS-P) to synthesize nano-composite cathodes for lithium and Li-Ion battery applications. Due to the peculiarities of this process we have been able to synthesize an intimately-mixed composite cathode formed by nanotubes of the well known active material LiFePO<sub>4</sub> interconnected with nanometer-size carbon particles. The average diameter of the nanotubes was in the range 400 nanometers to 50 nanometers according to the diameters of the pores of the Track-Etched Polycarbonate Membranes used as the starting templates.

The cathodes have been characterized by FE-SEM (Field-Emission Scanning Electron Microscopy), CV (Cycling Voltammetry), GC (Galvanostatic Cycling) and IS (Impedance Spectroscopy). Preliminary results show that these nano-composite tubular cathodes manifest improved kinetic performance when compared to the conventionally prepared micro-sized composite electrodes. With this respect it is worth to note, for example, that the differences between  $E_{pa}$ , the anodic peak potential, and  $E_{pc}$ , the cathodic peak potential, ( $\Delta E_p$ ), of the CV traces taken at 1 mV/s scan rate on nanocomposite cathodes were significantly lower than those of conventional micro-sized cathodes taken at 100  $\mu$ V/s scan rate

## Acknowledgements

This work has been performed in the frame of the research activities granted by US Army under contract number N68171-01-M5642

### References

- F. Croce, J. Hassoun, B. Scrosati, A. Deptula and L. Lada – *Electrochemistry and Solid State Letters* – In press.
- 2) A.K. Padhi, K.S. Nanjundaswamy and J.B. Goodenough, J. Electrochem. Soc., 144, 1188 (1997).
- 3) C.R. Martin, *Science*, Vol.266, 23 December (1994), 1961.
- 4) C.R.Martin, Chem. Mater. (1996) 8, 1739.
- 5) B.B Lakshimi, C.J. Patrissi and C. R. Martin, *Chem. Mater.* (1997), 9, 2544-2550.
- 6) C.J. Brinker, G. W. Scherer, *Sol-Gel Science*, Academic Press Inc., New York, 1990.
- F. Croce, F. Nobili, A. Deptula, W. Lada, R. Tossici, A. D'Epifanio, B. Scrosati, R. Marassi, *Electrochem. Comm.*, 1, 605 (1999)

<sup>•</sup> Presenting Author; Email:fausto.croce@uniroma1.it; on Sabbatical Leave to the Department of Chemistry, Center for Chemical Research at Bio/Nano Interface, University of Florida, Gainesville, Florida 32611, USA