# Optimisation of the Titanium oxysulfides (TiO<sub>y</sub>S<sub>z</sub>) performance used as positive electrode in lithium microbatteries

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### Introduction

An all solid-state-microbattery is defined as a stack of thin layers (~  $15\mu$ m) including the active part (electrodes and electrolyte), protective layers and current collectors. [1]

 $LiCoO_2$ ,  $LiMn_2O_4$  and  $V_2O_5$  are commonly used as positive electrode material. As reported in the literature,  $LiCoO_2$  and  $LiMn_2O_4$  operate reversibly around 4  $V/Li^+/Li$ . [2]

In this work, the stack Li/LiPON/TiOS is studied. Titanium oxysulfide thin films  $(TiO_yS_z)$  are used as positive electrode material. This material was selected because of their specific potential (reversibly insert of lithium below 3V vs. Li<sup>+</sup>/Li) well adapted for the considered application.

Correlation between microstructure, chemistry and performance of the active material is reported.

## Experimental

Thin films of  $TiO_yS_z$  were deposited by sputtering process (PVD) on 8" Si wafer. Power,  $H_2S$  flow rate and deposition temperature were modified to optimized  $TiO_yS_z$  performances.

 $TiO_yS_z$  deposition rate and microstructure were determinate by weighing and SEM analyses. The electrochemical properties were measured by galvanostatic cycling and EIS. The chemical composition was studied by X-Ray Photoelectron Spectroscopy.

#### **Results and discussion**

The increase of the deposition temperature, from room temperature to 150°C, improves the capacity and the fading rate. The new material shows an excellent cycle life with fading rate of -0.05% per cycle and a capacity of around 70  $\mu$ A.h.cm<sup>-2</sup>. $\mu$ m<sup>-1</sup> between 1V and 3V vs. Li<sup>+</sup>/Li (fig.1).

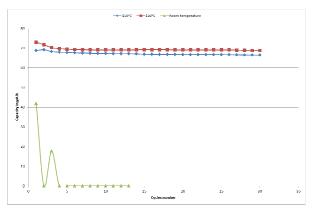


Figure 1: Evolution of the capacity with deposition temperature over 30 cycles, 150°C (red and blue curves), room temperature (green curve)

The SEM micrograph of the cross section shows a dense TiOS layer (fig.2).

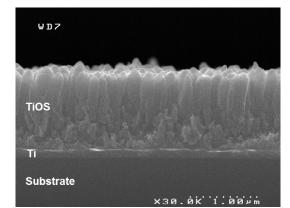


Figure 2: Cross section SEM micrograph of thin film of Ti/TiOS

#### Conclusion

The TiO<sub>y</sub>S<sub>z</sub> material is suitable to be used as positive electrode in microbattery. The three parameters power, H<sub>2</sub>S flow rate and deposition temperature allowed the deposition of TiO<sub>y</sub>S<sub>z</sub> with a capacity of 70  $\mu$ Ah cm<sup>-2</sup>  $\mu$ m<sup>-1</sup> and an excellent cycle life.

#### References

[1] B. Pecquenard, F. Le Cras, M. Martin, P. Vinatier, A. Levasseur, R. Salot, « Microbatteries - Microsources d'énergie en couches minces », Techniques de l'ingénieur, reference D3342, 10 mai 2009

[2] B. Fleutot, B. Pecquenard, F. Le Cras, B. Delis, H. Martinez, L. Dupont, D. Guy-Bouyssou « Characterization of all-solid-state Li/LiPONB/TiOS microbatteries produced at the pilot scale», Journal of Power Sources, 196, 2011, pp 10289–10296