



## Plasma assisted RF sputtering of lithium phosphorous oxynitride thin films for all-solid-state lithium ion batteries

Stamate, Eugen; Christiansen, Ane Sælland; Holtappels, Peter

*Publication date:*  
2013

[Link back to DTU Orbit](#)

*Citation (APA):*

Stamate, E., Christiansen, A. S., & Holtappels, P. (2013). *Plasma assisted RF sputtering of lithium phosphorous oxynitride thin films for all-solid-state lithium ion batteries*. Abstract from 5th International Symposium on Advanced Plasma Science and its Applications for Nitrides and Nanomaterials, Nagoya, Japan.

---

### General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

# Plasma assisted RF sputtering of lithium phosphorous oxynitride thin films for all-solid-state lithium ion batteries

E. STAMATE\*, A. CHRISTIANSEN, AND P. HOLTAPPELS

DEPARTMENT OF ENERGY CONVERSION AND STORAGE, TECHNICAL UNIVERSITY OF DENMARK

Frederiksborgvej 399, Roskilde - 4000, Denmark

phone: +45-4677 4562, fax: +45-4677 4565

e-mail: eust@dtu.dk

## 1. Introduction

Solid-state thin film batteries based on lithium phosphorous oxynitride (Lipon) are a strong candidate for compact and high power density sources needed for various portable electronic devices [1]. A rechargeable thin film battery electrolyte needs to show high ionic conductivity, to be free of porosity or cracks, and not to decompose in contact with the anode. So far, several thin film deposition techniques have been used to produce Lipon films with different properties [2]. Magnetron sputtering with a deposition rate below 3 nm/min shows good electrochemical properties. Despite extensive research the role of nitrogen is not yet well understood [3]. The aim of this work is to correlate the plasma and thin-films properties employing detailed plasma diagnostics by mass spectrometry, optical emission spectroscopy and probes in a plasma assisted magnetron sputtering device.

## 2. Experimental setup and discussions

Experiments are performed in a 3×4 matrix configuration of distributed ECR plasma cells placed at the top of a 40×40×40 cm vacuum chamber (see Fig. 1). The cells can produce a uniform plasma in a pressure range from 10 down to 0.1 mTorr. A 2 inch magnetron sputtering cathode is placed on the lateral side facing the head of a Hiden mass spectrometer. A Langmuir probe and an optical fiber are inserted perpendicular to the direction of the mass spectrometer and cathode as to measure plasma parameters and optical emission spectra. The target made of  $\text{Li}_3\text{PO}_4$  is provided by Kurt Lesker®. Lipon films are deposited onto Au-coated silicon substrates using different values for  $\text{N}_2$  flow and RF power in a controllable nitrogen

flow. A ceramic mask is used to delineate deposition areas of 10×10 mm. The substrate was not heated nor biased during the sputtering. The typical deposition rate was 2.4 nm/min for 100 W RF power for a total thickness of about 1  $\mu\text{m}$ . A thin film of silver was deposited on top of the Lipon.

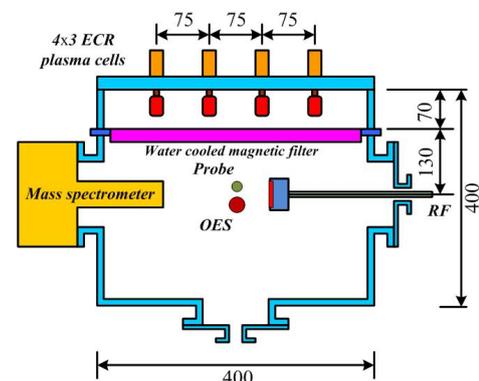


Fig. 1 RF magnetron sputtering assisted by 3×4 matrix ECR plasma cells.

The ionic conductivity was measured by impedance spectroscopy and the microstructure was investigated using focused ion beam scanning electron microscopy. The highest conductivity of  $2.1 \times 10^{-6}$  S/cm was obtained at 5 mTorr for 100 W RF power, where the dissociation degree of nitrogen was also highest both detected by mass spectrometry and optical emission. Further investigations are necessary in order to correlate the measured parameters with the role of nitrogen in controlling the ionic conductivity.

## References

- [1] J. Schwenzel, V. Thangadurai, W. Weppner, *J. Power. Sources* 154 (2006) 232
- [2] Y. G. Kim and H. N. G. Wadley, *J. Vac. Sci. Technol. A*, 26 (2008) 174
- [3] M. Munoz, *J. Power. Sources* 198 (2012) 432