Department of Applied Physics and Science Education

Atomic layer deposition of LiPON using a novel lithium precursor

Low temperature process development and thin film characterization

by

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MSc THESIS

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Public summary

Society is shifting towards an energy system based on green energy sources, the intermittency of these sources, however, leads to a demand for energy storage. Amongst the energy storage technologies, Li-ion batteries (LIBs) emerge as one viable candidate, for both stationary and mobile applications, such as electric vehicles (EVs). Therefore there is an increasing demand for batteries with high energy and power density. Material science is key to high energy density and safe batteries. Presently, there are developments towards high voltage cathodes, high capacity anodes and solid state electrolytes (SSEs). Equally strategic is the research on the development of electrochemically and mechanically stable electrode/electrolyte interfaces. One interesting material is lithium phosphorus oxynitride (LiPON), which can serve as a SSE for micro storage applications, since its ionic conductivity is limited. Additionally, it can serve as an interface layer, due to its wide electrochemical stability window, on next generation electrode materials, such as Li-metal. These applications require LiPON to be applied as a conformal thin film. A technique that is very suitable for these deposition requirements is atomic layer deposition (ALD), due to its high conformality, uniformity and growth control. Most LiPON processes reported in literature use deposition temperatures of > 200 °C and are therefore not compatible with Li-metal.

In this thesis a process is developed for depositing LiPON using ALD at a relatively low deposition temperature of 150°C, making the recipe compatible with Li-metal. A novel lithium precursor, called Lider, is used in combination with trimethylphosphate (TMPO), a phosphate precursor. First LiPO films are fabricated with a stoichiometry of $Li_{2.2}PO_4$ and undetectable C at.%, measured using X-ray photoelectron spectroscopy (XPS). The reason for the low C at.% in the LiPO films was investigated and it was found that TMPO reacts with a Li_2CO_3 surface, similar to trimethylaluminum, which results in the removal of carbonate impurities. To obtain LiPON films, N doping is achieved by means of a N_2 plasma. The grown LiPON films show a stoichiometry of $Li_2PO_{2.7}N_{0.6}$ and the level of N incorporation is up to 7.5 at.%. The shape of the N1s XPS spectrum is very promising regarding the ionic conductivity of the films. These results show that it is possible to deposit LiPON films at 150°C using ALD. The developed process therefore enables the deposition of LiPON thin films as coatings on Li-metal anodes or as SSE in thin film microbatteries.