

Tuesday Afternoon, November 11, 2014

Thin Film

Room: 307 - Session TF+EN+PS-TuA

ALD for Energy

Moderator: Erwin Kessels, Eindhoven University of Technology, Netherlands

2:20pm **TF+EN+PS-TuA1 Li-Based ALD Solid Electrolytes for Beyond-Li-Ion Batteries, Alexander Kozen***, A.J. Pearse, M.A. Schroeder, C. Liu, M. Noked, C.F. Lin, G.W. Rubloff, University of Maryland, College Park

Solid Li-based inorganic electrolytes offer profound advantages for energy storage in 3-D solid state batteries: (1) enhanced safety, since they are not flammable like organic liquid electrolytes; and (2) high power and energy density since the solid electrolyte can support interdigitated nanostructured electrodes, avoiding binders, separators, and much larger spacing (tens of mm's) between fully separated electrodes. The quality of thin solid electrolytes – even in planar form – is currently a major obstacle to solid state batteries[1] restricting electrolyte thickness to >100 nm to control electronic leakage, consequently slowing ion transport across the electrolyte and impeding interdigitated 3-D nanostructure designs that offer high power and energy. Furthermore, the ion-conducting, electron-insulating properties of solid electrolytes are promising for their use as passivation or protective layers on metal anodes (Li, Na, Mg) and on cathodes in proposed “beyond-Li-ion” battery configurations such as Li-O₂ and Li-S.

Atomic layer deposition (ALD) is well suited to the challenge of solid electrolytes, providing ultrathin, high quality films with exceptional 3-D conformality on the nanoscale. We have developed ALD processes for Li₂O, Li₃PO₄, and LiPON from LiO^tBu, H₂O, and N₂, exploiting spectroscopic ellipsometry, downstream mass spectrometry, and XPS surface analysis, all *in-situ*. Post-ALD XPS reveals for the first time carbon-free electrolytes and their intrinsic surface chemistry. E.g., ALD Li₂O grown at 250C is reversibly transformed to LiOH upon exposure to H₂O, but transforms back upon annealing. LiOH is completely and irreversibly converted to Li₂CO₃ by CO₂ exposure. These kinds of observations are essential to developing process sequences for fabricating 3-D solid batteries.

We then demonstrate the impact of this solid electrolyte synthesis in several examples. For solid state batteries, we employ the electrolytes in planar and nanostructured battery configurations to determine their Li diffusivity and electrochemical performance. For beyond-Li-ion configurations with organic electrolytes, we show the use of ALD Li₂O at controlled mass loading in high aspect ratio Li-O₂ cathodes to elucidate the Li-O₂ charging chemistry, and we demonstrate the use of the ALD solid electrolytes in passivating Li anodes in Li-S batteries.

[1] D. Ruzmetov, V. P. Oleshko, P. M. Haney, H. J. Lezec, K. Karki, K. H. Baloch, A. K. Agrawal, A. V. Davydov, S. Krylyuk, Y. Liu, J. Huang, M. Tanase, J. Cumings, and A. A. Talin, “Electrolyte Stability Determines Scaling Limits for Solid-State 3D Li Ion Batteries,” *Nano Lett*, vol. 12, no. 1, pp. 505–511, Jan. 2012.

2:40pm **TF+EN+PS-TuA2 Engineering Lithium-Containing Ionic Conductive Thin Films by Atomic Layer Deposition for Lithium-ion Battery Applications, Jea Cho, T. Seegmiller, J. Lau, L. Smith, J. Hur, B. Dunn, J.P. Chang**, University of California at Los Angeles

Lithium (Li)-ion batteries have drawn much attention for their outstanding performance in portable electronics applications. These batteries have the potential to function as miniaturized power sources for microelectromechanical (MEMS) devices through the fabrication of 3-dimensional configurations. To fabricate a fully functional 3D Li-ion microbattery, however, an ultra-thin and highly conformal electrolyte layer is required to coat the 3D electrodes. The solid oxide Li-ion conductor, lithium aluminosilicate (Li_xAl_ySi_zO, LASO), synthesized by atomic layer deposition (ALD) is a promising electrolyte material for 3D battery applications owing its adequate ionic conductivity as well as improved electrode stability.

The self-limiting characteristic of ALD allows for precise control of thickness and composition of complex oxides and results in a highly conformal and pinhole-free coating even on highly complex structures such as high aspect ratio 3D electrodes. The metal precursors, lithium t-butoxide (LTB), trimethylaluminum (TMA), tris(tert-butoxy)silanol (TTBS), and

tetraethylorthosilicate (TEOS) were used to form Li_xAl_ySi_zO via ALD. *In-situ* FTIR was implemented to study the incubation time and growth mechanisms for each oxide deposited on the other to improve the controllability of the films. *In-situ* FTIR studies revealed that the growth mechanism of silicon oxide is strongly affected by the underlying oxide layer, exhibiting different surface reaction mechanisms during the incubation stage.

Li-ion conductivities and the activation energy for conduction of as-deposited LASO/LAO/LSO films were determined for different lithium contents and film thickness. The LASO ALD coating on 3D carbon array posts were confirmed to be conformal and uniform using transmission electron microscopy (TEM) imaging. A Li-ion half-cell consisting of LASO coated on 3D carbon array electrode showed reversible electrochemical behavior. Lithiation cycling tests of thin LASO/LAO/LSO films were found to be functions of both composition and thickness. The reversibility and kinetics of insertion as well as the effect on the cycling stability from the direct deposition of LASO/LAO/LSO on potential anode materials, SiNWs were also investigated using *in-situ* TEM observations during lithiation.

3:00pm **TF+EN+PS-TuA3 Applications of ALD for Li ion Batteries and Low Temperature Fuel Cells, Xueliang (Andy) Sun**, University of Western Ontario **INVITED**

Atomic layer deposition (ALD) is a novel and unique coating technique with many applications in energy storage and conversion [1]. In this talk, I will present our recent work on exploring the applications of atomic layer deposition (ALD) in both fuel cells and Li ion batteries [2-7].

In the first part, we will report of use of ALD for Pt catalysts used in low temperature fuel cells. In particular, atomic Pt or clusters prepared by ALD show ten times higher methanol oxidation properties compared with ETK commercially-used catalysts [2].

In the second part, we will focus on employing ALD as a surface-modification method to enhance the performance of LIBs. Different materials for surface-modification (such as Al₂O₃, ZrO₂, TiO₂ and AlPO₄) [3,4] were first developed by ALD. Then systemic studies were carried out by using those materials to modify the anode (Li₄Ti₅O₁₂, SnO₂) [5] and the cathode (commercial LiCoO₂, NMC) [6]. The effects of different coating materials on the LIB performance of the anode and cathode were investigated in details. In addition, the potential application of ALD as a powerful technique for preparing solid-state electrolyte will be demonstrated [7]. We will discuss further development of ALD for fuel cells and Li ion batteries.

Reference:

- [1] X. Meng, X.-Q. Yang, X. Sun. *Adv. Mater.* 2012, 24, 3589-3615.
- [2] S. Sun , G. Zhang, N. Gauquelin, N. Chen, J. Zhou, S. Yang, W. Chen, X. Meng, D. Geng, M. Banis, R. Li,
- S. Ye, S. Knights, G. Botton, T.-K. Sham, X. Sun, *Scientific Reports* 3 (2013) 1775.
- [3] J. Liu, X. Meng, Y. Hu, D. Geng, M.N. Banis, M. Cai, R. Li, X. Sun. *Carbon* 2013, 52, 74-82.
- [4] J. Liu, Y. Tang, B. Xiao, T.K. Sham, R. Li, X. Sun. *RSC Adv.* 2013, 3, 4492-4495.
- [5] X. Li, X. Meng, J. Liu, D. Geng, Y. Zhang, M. Banis, Y. Li, R. Li, X. Sun, M. Cai, M. Verbrugge, *Adv. Funct. Mater.* 22 (2012) 1647-1654.
- [6] X. Li, J. Liu, M. Banis, A. Lushington, R. Li, M. Cai, X. Sun, *Energy Environ. Sci.* 7 (2) (2014) 768-778
- [7]. J. Liu, M. Banis, X. Li, A. Lushington, M. Cai, R. Li, T.-K. Sham, X. Sun, *J. Phys. Chem. C* 117(2013) 20260-20267

4:20pm **TF+EN+PS-TuA7 ALD for a High Performance “All-in-One” Nanopore Battery, Chanyuan Liu, X. Chen, E. Gillette, A.J. Pearse, A.C. Kozen, M.A. Schroeder, K. Gregorczyk, S.B. Lee, G.W. Rubloff**, University of Maryland, College Park

A self-aligned nanostructured battery fully confined within a single nanopore presents a powerful platform to determine the performance and cyclability limits of nanostructured storage devices. We have created and evaluated such structures, comprised of nanotubular electrodes and electrolyte confined within anodic aluminum oxide (AAO) nanopores as “all-in-one” nanopore batteries. The nanoelectrodes include metal (Ru or Pt) nanotube current collectors with crystalline V₂O₅ storage material on top of them, penetrating part way into the AAO nanopores to form a symmetric full storage cell, with anode and cathode separated by an electrolyte region.

* TFD James Harper Award Finalist

The unprecedented thickness and conformality control of atomic layer deposition (ALD) and the highly self-aligned nanoporous structure of anodic aluminum oxide (AAO) are essential to enable fabrication of precision, self-aligned, regular nanopore batteries, which display exceptional power-energy performance and cyclability when tested as massively parallel devices (~ 2 billion/cm²), each with $\sim 1 \mu\text{m}^3$ volume (~ 1 fL).

To realize these “all-in-one” nanopore batteries, we focused on the precise control of Ru and Pt thin film conformality inside very high aspect ratio (300:1) AAO nanopores by thermal ALD process. 7.5 nm thick Ru and Pt are optimized to be 15 μm deep at both sides of 50 μm long AAO pores in order to provide fast electron transport to overlying V₂O₅ at both anode and cathode sides, while keeping them spatially and electrically isolated. Active storage layers of 23 nm thick crystalline V₂O₅ were deposited inside the metal nanotubes to form core-shell nanotubular structures at low temperature (170 °C) using O₃ as the oxidant, with <001> direction perpendicular to tube surface and RMS roughness ~ 4 nm. Then the V₂O₅ was prelithiated at one end to serve as anode while pristine V₂O₅ without Li at the other end served as cathode, enabling the battery to be cycled between 0.2 V and 1.8 V and to achieve full theoretical Faradaic capacity of the V₂O₅. Capacity retention of this full cell at high power (relative to 1 C rates) is 95% at 5 C and 46% at 150 C rates (i.e., 24 sec charge/discharge time). At 5 C rate (12 min charge-discharge cycle), 81.3% capacity remains after 1000 cycles. These performance metrics are exceptional, exceeding those of most prototypes reported in the literature. These results demonstrate the promise of ultrasmall, self-aligned/regular, densely packed nanobattery structures as a building block for high performance energy storage systems.

4:40pm TF+EN+PS-TuA8 Pseudocapacitive Manganese Oxide Grown by Atomic Layer Deposition, Matthias Young, C.D. Hare, A.S. Cavanagh, C.B. Musgrave, S.M. George, University of Colorado, Boulder

Pseudocapacitive supercapacitors are a class of energy storage materials that are midway between lithium ion batteries and capacitors in terms of both power and energy densities. Manganese oxide is a well-known pseudocapacitive material with particular appeal due to its earth abundance and low cost. In previous work, we have demonstrated that MnO ALD produced using bis(ethylcyclopentadienyl)manganese (Mn(CpEt)₂) and water can be electrochemically oxidized to produce pseudocapacitive MnO₂ in aqueous electrolytes. However, recent results have shown that the electrochemical oxidation of MnO ALD films results in partial dissolution and delamination. To avoid these problems, we have worked to grow pseudocapacitive MnO₂ by ALD that requires no post-processing. We have grown manganese oxide ALD films using ozone as the coreactant with Mn(CpEt)₂. We have also used intermediate ozone doses during Mn(CpEt)₂ and water exposures during ALD growth. The use of ozone results in more oxidized manganese oxide films. Another issue is that the alpha-MnO₂ crystal structure of MnO₂ which exhibits high pseudocapacitance contains open channels that are only stable in the presence of cations such as Na⁺ or K⁺. Consequently, directing the ALD growth toward alpha-MnO₂ pseudocapacitive crystal structures requires the incorporation of an alkali metal into the MnO₂ ALD films.

5:00pm TF+EN+PS-TuA9 Excellent Chemical Passivation of p⁺ and n⁺ Surfaces of Silicon Solar Cells by Atomic Layer Deposition of Al₂O₃ and SiO₂/Al₂O₃ Stacks, Bas van de Loo, H.C.M. Knoops, Eindhoven University of Technology, Netherlands, G. Dingemans, ASM, Netherlands, I.G. Romijn, ECN Solar Energy, Netherlands, W.M.M. Kessels, Eindhoven University of Technology, Netherlands

Thin films of Al₂O₃ provide excellent passivation of heavily p-doped (p⁺) silicon surfaces and are therefore often applied in silicon solar cells to reach high efficiencies. The high level of passivation by Al₂O₃ can be attributed to its low interface defect density and high negative fixed charge density Q_f. However, the negative fixed charge density of Al₂O₃ can be detrimental for the passivation of n⁺ surfaces [1]. Furthermore, in advanced cell architectures such as interdigitated back-contact (IBC) solar cells, both n⁺ and p⁺ surfaces are adjacent and are preferably passivated simultaneously. To this end, we systematically study the surface passivation by SiO₂/Al₂O₃ stacks prepared by atomic layer deposition (ALD), which exhibit excellent chemical passivation while the effective fixed charge density can be tuned to zero by carefully tuning the SiO₂ thickness.

Al₂O₃ and SiO₂/Al₂O₃ film stacks with varying ALD SiO₂ thickness (0-12 nm) were prepared by plasma-enhanced ALD at 200 °C, with H₂Si(N(C₂H₅)₂)₂ and Al(CH₃)₃ as metal-organic precursors and O₂ plasma as oxidant. Moreover, a SiO₂ ALD process using ozone was developed as this oxidant is more suitable for batch ALD. The relevant process parameters for surface passivation, such as ozone exposure time, were identified. The passivation of n⁺ and p⁺ doped surfaces was studied in detail, and results were compared with industrial passivation schemes, including PE-CVD SiN_x and similar SiO₂/Al₂O₃ stacks from a high-volume manufacturing ALD batch reactor.

A superior level of passivation of n⁺ surfaces (R_{sheet} = 100 Ω/sq) was obtained by SiO₂/Al₂O₃ stacks as compared to single layer Al₂O₃, significantly reducing the recombination current density (J₀) from (81±10) to (50±3) fA/cm². On p⁺ surfaces (R_{sheet} = 60 Ω/sq), J₀ increases with increasing SiO₂ thickness. The results can be explained by an excellent level of chemical passivation, combined with a strongly reduced negative fixed charge density when increasing the SiO₂ thickness. To fully exploit the virtues of ALD, the concept of using SiO₂/Al₂O₃ stacks for the passivation of both the n⁺ and p⁺ doped surfaces in a single deposition run was demonstrated on (completed) n-type bifacial solar cells, reaching conversion efficiencies >19%. The results are promising for IBC solar cells, where n⁺ and p⁺ surfaces are adjacent and care must be taken to achieve a low surface recombination, high shunt resistance and industrial feasibility.

[1] B. Hoex *et al.*, *Phys. status solidi - Rapid Res. Lett.*, vol. 6, no. 1, pp. 4-6, (2012).

5:20pm TF+EN+PS-TuA10 Opportunities for Transparent Conductive Oxides Prepared by ALD for Silicon Heterojunction Solar Cells, Bart Macco, S. Smit, Y. Wu, D. Vanhemel, W.M.M. Kessels, Eindhoven University of Technology, Netherlands

In silicon heterojunction (SHJ) solar cells, transparent conductive oxides (TCOs) serve as the top window layer which provides lateral charge transport to the metal contacts whilst maintaining a high optical transparency. Commonly-employed TCO materials include Sn-doped indium oxide (In₂O₃:Sn), Al-doped zinc oxide (ZnO:Al) and more recently also H-doped indium oxide (In₂O₃:H)¹, which are typically deposited by sputtering. In this work, atomic layer deposition (ALD) is explored as an alternative deposition technique for the abovementioned materials. Three salient features of the ALD process will be addressed. Firstly, the applicability of these ALD TCOs is evaluated in terms of their optoelectronic performance. It is shown that through controlled ALD doping cycles the carrier density can be accurately tuned and a low resistivity (<0.5 mΩcm) required for SHJ solar cells can be obtained. Secondly, it is shown that a thermal ALD process does not induce damage to the underlying a-Si:H passivation layers found in a SHJ solar cell. This is a distinct advantage over the conventional sputtering technique, in which plasma-related (UV, ions) damage is known to reduce the passivation level of the a-Si:H layers.² This perk of ALD is put to use in bilayers of ALD ZnO:Al/sputtered In₂O₃:Sn, where a thin ALD TCO layer (<15 nm) can very effectively protect the a-Si:H layers from sputter damage. TEM and *in-situ* spectroscopic ellipsometry measurements show that the protective properties are strongly correlated with the TCO surface coverage, as the initial ALD TCO growth on the a-Si:H layer suffers from a nucleation delay and associated island-like growth.³ Finally, the accurate control over the doping (profile) of the TCO offered by ALD opens up ways to optimize the band alignment of a SHJ solar cell. At the interface of the TCO and the p-type a-Si:H, a high doping of the TCO is unfavorable for the band alignment and results in a reduced fill-factor.³ On the other hand, the conductivity requirement of the TCO sets a lower bound to the doping level. In this respect, graded doping of the TCO by ALD allows for effective decoupling of the conductivity requirements of the TCO with the optimization of the interface contact formation.

¹ Barraud *et al.*, *Solar Energy Materials and Solar Cells*, **115**, 151–156 (2013)

² Demarex *et al.*, *Applied Physics Letters*, **101**, 171604 (2012)

³ Macco *et al.*, *Applied Physics Letters* (submitted)

5:40pm TF+EN+PS-TuA11 Study of the Surface Passivation Mechanism of c-Si by Al₂O₃ using In Situ infrared spectroscopy, R.P. Chaukulkar, Colorado School of Mines, W. Nemeth, A. Dameron, P. Stradins, National Renewable Energy Laboratory, Sumit Agarwal, Colorado School of Mines

The quality of Si surface passivation plays an integral role in the performance of c-Si-based solar cells. Recently, Al₂O₃ films grown by atomic layer deposition (ALD) have been shown to be an effective passivant for c-Si surfaces with surface recombination velocities (S_{eff}) that are <5 cm/s. The chemical passivation of the c-Si surface via Al₂O₃ is achieved by a reduction in the defect density at the interface, while field-effect passivation is attributed to the fixed negative charge associated with the Al₂O₃ films. However, a post-deposition annealing step is required to achieve this high level of passivation. We have investigated the mechanism of chemical passivation during the annealing step using *in situ* attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy. Specifically, we have studied the role of residual H- and O-atom migration from the ALD Al₂O₃ films to the c-Si/Al₂O₃ interface. Using Al(CH₃)₃ and O₃ as the ALD precursors, Al₂O₃ films were deposited directly onto high-lifetime float-zone c-Si internal reflection crystals (IRCs) followed by thermal annealing at 400 °C in different atmospheres. Specifically, we have used D-terminated c-Si IRCs to differentiate the residual H atoms that may

migrate from ALD Al_2O_3 films versus the residual D atoms present at the $\text{Al}_2\text{O}_3/c\text{-Si}$ interface after ALD. Within the sensitivity of the ATR-FTIR spectroscopy setup of $\sim 10^{12} \text{ cm}^{-2}$ for Si-H bonds, we do not detect any migration of H from Al_2O_3 to the $c\text{-Si}$ interface. Therefore, we conclude that the migration of O, and the subsequent restructuring of the interface during the annealing step, primarily contributes towards the chemical passivation of the $\text{Al}_2\text{O}_3/c\text{-Si}$ interface. The ATR-FTIR spectroscopy measurements are complemented by the minority carrier lifetime, interface defect density, and built-in charge density measurements on $\text{SiO}_2/\text{Al}_2\text{O}_3$ stacks on $c\text{-Si}$, which enable us to isolate chemical passivation from field-effect passivation. The stacks were annealed in different atmospheres to better understand the role of O versus H atoms in the chemical passivation mechanism.

We gratefully acknowledge the support from the NCPV Fellowship Program and U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, under Contract No. DE-AC36-08-GO28308 with the National Renewable Energy Laboratory.

6:00pm TF+EN+PS-TuA12 Low Temperature Plasma-assisted Atomic Layer Deposition of TiO_2 Blocking Layers for Flexible Hybrid Mesoscopic Solar Cells, V. Zardetto, Eindhoven University of Technology, Netherlands, F. di Giacomo, T.M. Brown, A. di Carlo, A. D'Epifanio, S. Licoccia, University of Rome "Tor Vergata", Italy, W.M.M. Kessels, *Mariadriana Creatore*, Eindhoven University of Technology, Netherlands

Atomic Layer Deposition (ALD) is widely acknowledged in the field of $c\text{-Si}$ and thin film PV technologies, for the fabrication of ultra-thin, uniform and conformal layers.[1] Thermal ALD has been applied also in the case of more challenging interfaces, e.g. dye-sensitized solar cells (DSCs) and the novel hybrid organo-lead-halide perovskite solar cells. Particularly, TiO_2 blocking layers have been developed on glass/TCO substrates with the aim of decreasing the charge recombination processes at the interface between the ITO and the mediator. Recently, we have explored the benefit of plasma-assisted ALD (PA-ALD) in terms of low temperature processing applied to flexible DSCs for the development of highly transparent Pt counterelectrodes on ITO/PEN. [2] In this work, we further explore PA-ALD for the deposition of ultra-thin, highly compact TiO_2 blocking layers on ITO-polymer substrates for DSCs and perovskite solar cells. The layers were prepared in a remote plasma reactor (FlexALTM) at 150 °C using an heteroleptic alkylamido precursor $\text{Ti}(\text{Cp}^{\text{Me}})(\text{NMe}_2)_3$ alternated with an O_2 plasma. For DSCs with an iodide-based electrolyte, the introduction of the blocking layer is essential at low light intensity, in order to increase the indoor performance of the cell. It is found that the presence of ultra-thin (6 nm) TiO_2 layers slightly affects the performance of the cell under sun simulator, whereas it definitely improves the generated power (+40%) under low level illumination (300 lux). The blocking behaviour of the PA-ALD deposited TiO_2 towards the tri-iodide reduction has been investigated by electrochemical impedance spectroscopy and Tafel plot analysis. We pinpointed that an increase in the TiO_2 layer thickness above 6 nm leads to a decrease of the recombination processes at the TCO/electrolyte interface, as well as to a dramatic reduction of the electron collection at the TCO, accompanied by a decrease in cell performance. For mesostructured perovskite ($\text{CH}_3\text{NH}_3\text{PbI}_2\text{Cl}$ - based) solar cells, the application of a TiO_2 blocking layer is essential for the performance of the device, due the higher current exchange at the interface TCO-hole transport material, i.e. Spiro-OMeTAD, typically used in this architecture. The application of a 11 nm-thick TiO_2 layer resulted in an efficiency of 7.4%. In conclusion, ALD is a valid approach for controlling electrochemical charge-transfer processes in mesoscopic solar cells.

[1] J.A. van Delft, D. Garcia-Alonso, W. M. M. Kessels, *Semicond. Sci. Technol.*, 27, 74002 (2012)

[2] D. Garcia-Alonso, V. Zardetto, A.J.M. Mackus, F. De Rossi, M.A. Verheijen, T.M. Brown, W.M.M. Kessels, M. Creatore, *Adv. En. Mater.* 4, 1300831 (2014)

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