

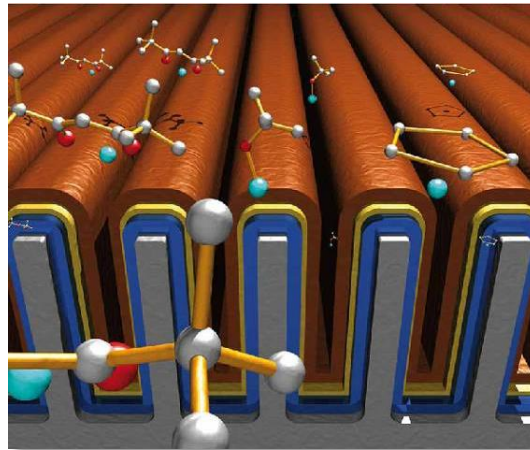
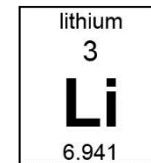


UiO : Centre for Materials Science and Nanotechnology

University of Oslo

Ola Nilsen

Atomic layer deposition for microbatteries



The abstract...

All **solid state microbatteries** can only be realised using pinhole free films, particularly for the electrolytes, where also thinner is better in terms of ionic conductivity. The atomic layer deposition (ALD) technique is identified as perhaps the enabling technology for realisation of such structures, also on 3D structures.

The current contribution will give an **overview of the present status** in deposition of materials for microbatteries by ALD, with highlights from both the anodes, electrolytes and cathodes, where **recent findings in pseudocapacitive behaviour of LiFePO_4** will be given. The ALD technique has traditionally been used for deposition of dielectric materials, also when it comes to ionic conductivities, while battery materials are ideally highly conducting with respect to Li-ions. This has led to **challenges in deposition processes containing lithium** where the growth perhaps no longer is strictly terminated by the surface reactions. What implications does this have on the ALD-growth?

¹ Author to whom any correspondence should be addressed.
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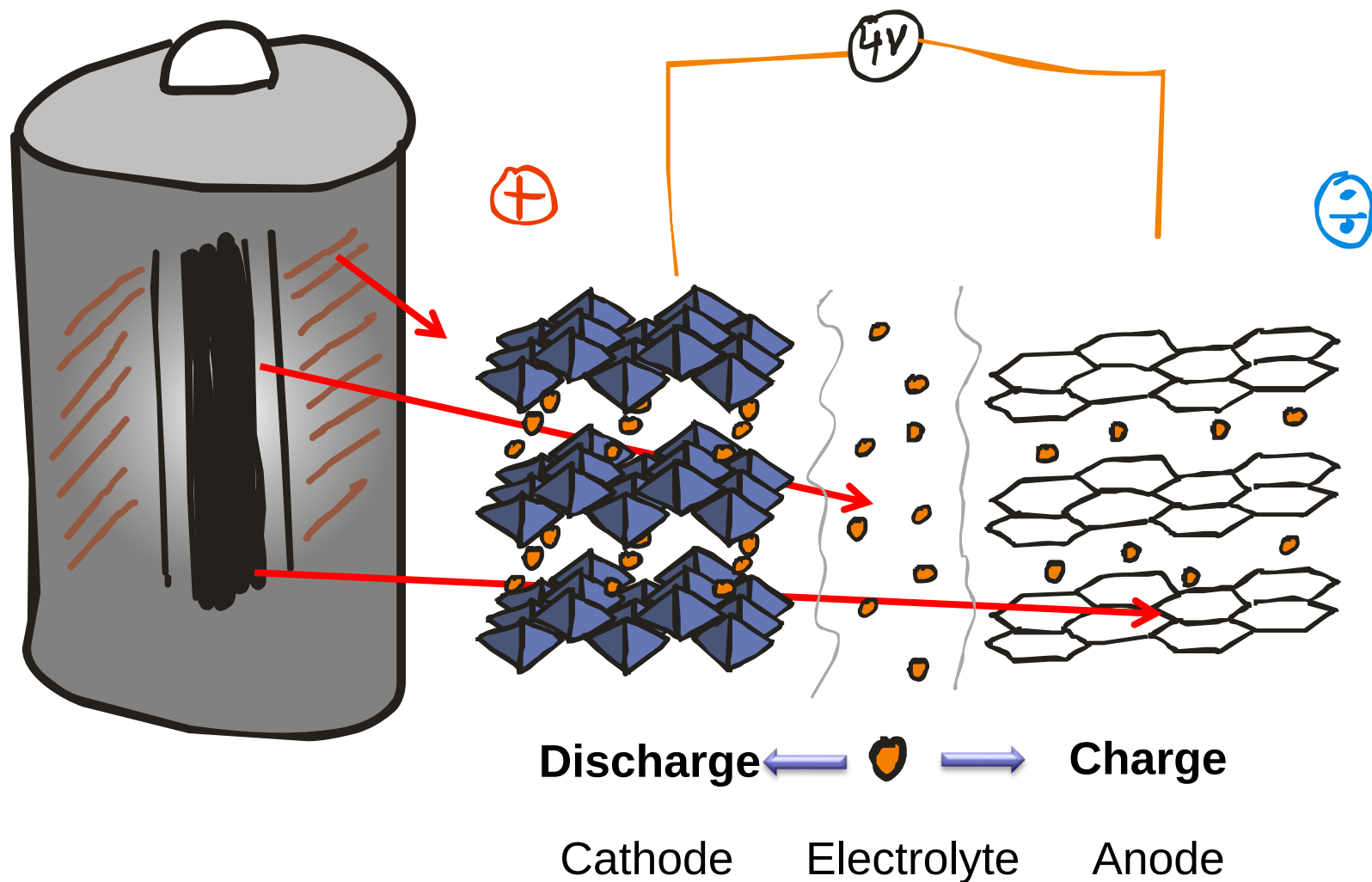
6 Infecting their enemy over other LIB cell construction, anode (negative), a lithium ion-selective electrode easily insulating conductive electrons between the electrodes can be a polymer, or an inorganic. LIB use liquid electrolyte such as LiDFOB, and they are dissolved in ethyl carbonate solution (dimethyl, diethyl ether) (e.g., EC, DMC). They are spontaneous due to their different potentials [7] rely on the situation.

The two electrodes chemistry governs the first commercial LIBs provide a capacity ranging from 100 mAh/g to 200 mAh/g storage. So far, most in terms of the are expected to be the construction of the cathodes is still expensive. The use of hybrid electrodes like V₆O₁₃/Uniformity suffer from (i.e., cost safety, cell large-discharge, and cycles, cathodes, etc.) [8]. More sophisticated temperature large-discharge cycles these requirements. Therefore, innovative new materials for the some nanostructured materials for electronic and their composites.

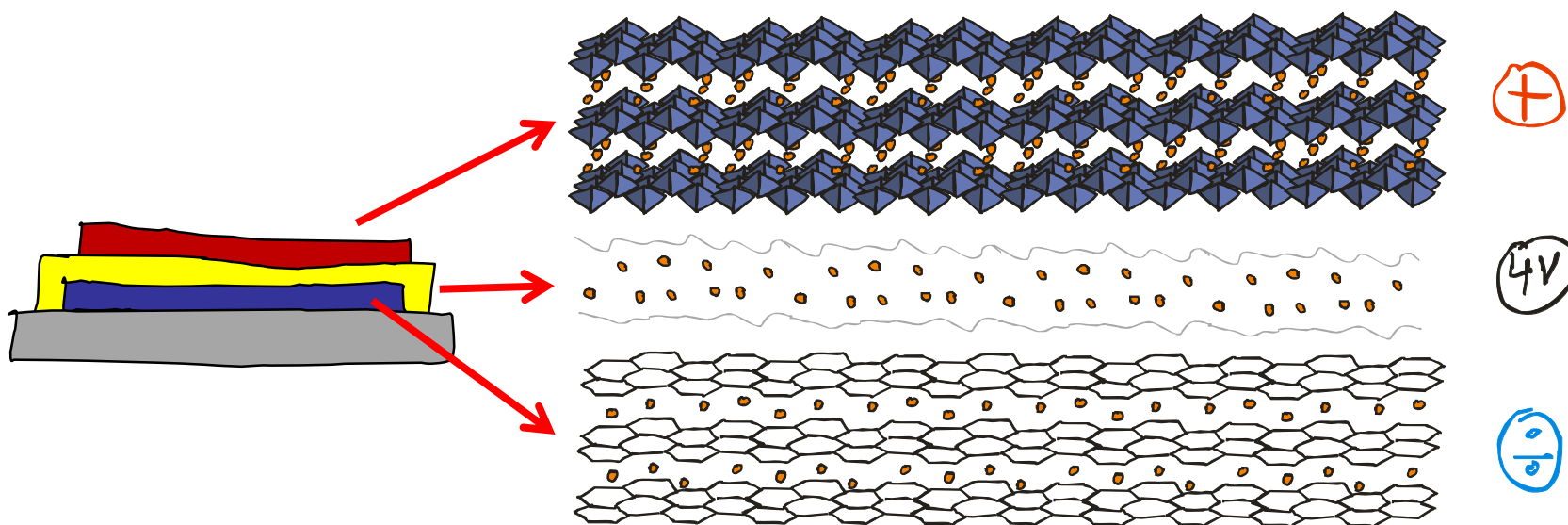
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T. Aaltonen, ..., H. Fjellvåg, *ECS Transactions* **41** (2011) 331. X. Meng, ..., X. Sun, *Advanced Materials*, **24** (2012) 3589.

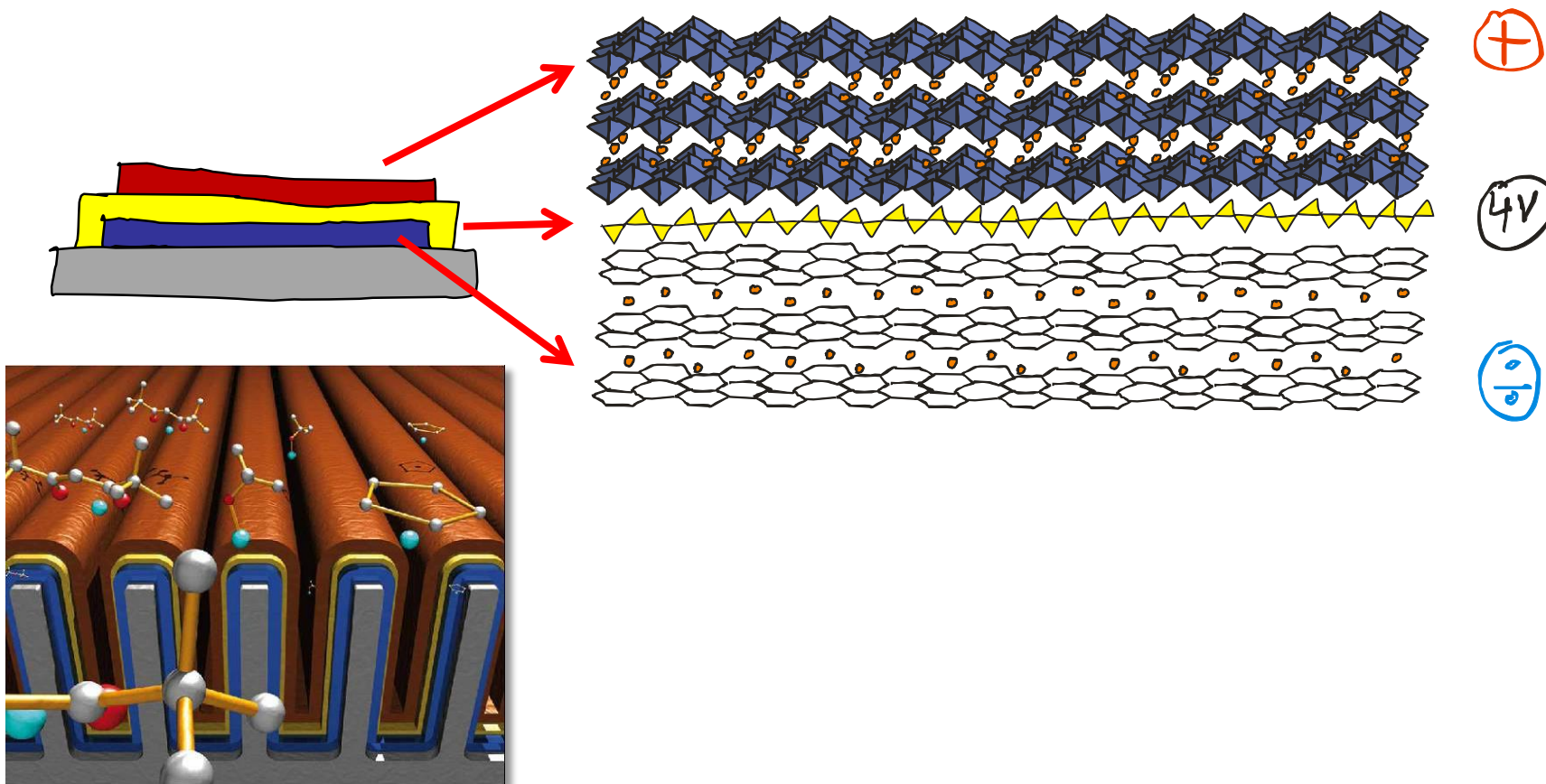
Li-battery



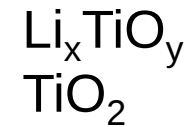
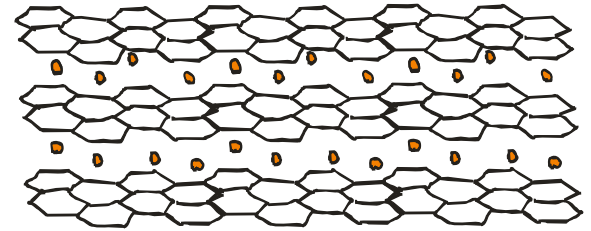
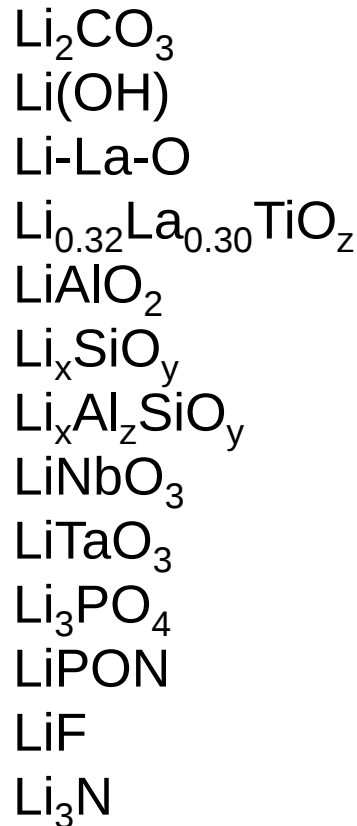
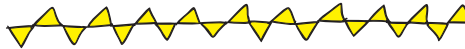
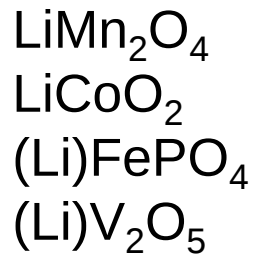
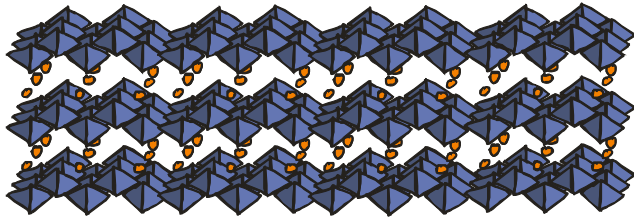
Li-film battery



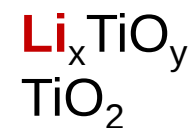
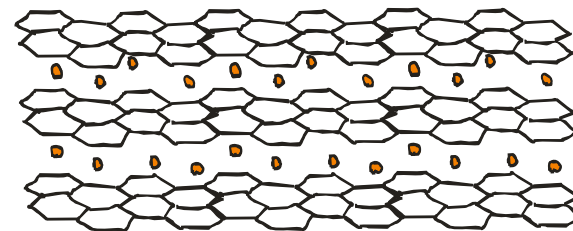
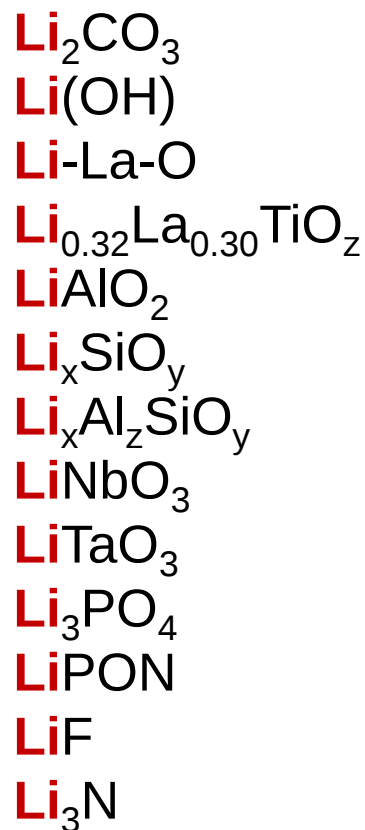
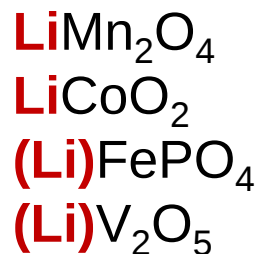
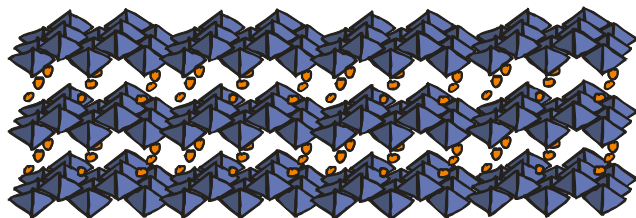
Li-film battery

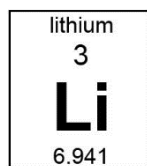


Li-film battery

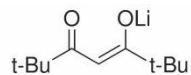


Li-film battery

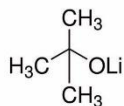




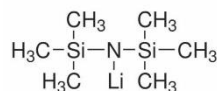
- precursors



Li(thd)
Lithium (2,2,6,6-tetramethyl-3,5-heptanedionato)



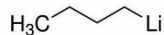
Li(O^tBu)
Lithium *tert*-butoxide



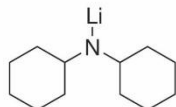
LiHMDS
Lithium hexamethyldisilazane
Lithium bis(trimethylsilyl)amide



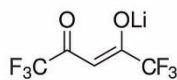
Li(Cp)
Cyclopentadienyllithium



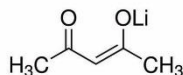
BuLi
Butyllithium



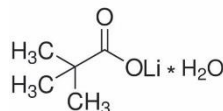
Lithium dicyclohexylamide



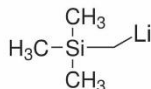
Li(hfac)
Lithium(1,1,1,5,5,5-hexafluoro-2,4-pentanedionato)
Lithium hexafluoroacetylacetonate



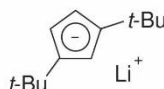
Li(acac)
Lithium acetylacetonate



Li(Piv)(H₂O)
Lithium pivalate *H₂O



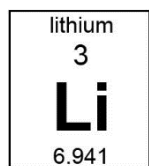
Li(CH₃SiMe₃)
Lithium trimethylsilane



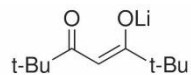
Li(^tBu2Cp)
Lithium Di-*tert*-butylcyclopentadiene



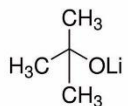
M. Putkonen, T. Aaltonen, M. Alnes, T. Sajavaara, O. Nilsen, and H. Fjellvåg, *J. Mater. Chem.* **19**, 8767 (2009).



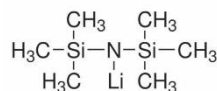
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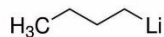
Li(O^tBu)
Lithium *tert*-butoxide



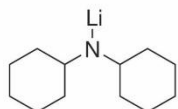
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Lithium hexamethyldisilazane
Lithium bis(trimethylsilyl)amide



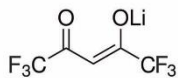
Li(Cp)
Cyclopentadienyllithium



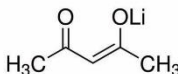
BuLi
Butyllithium



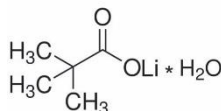
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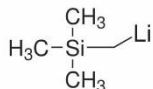
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Lithium hexafluoroacetylacetonate



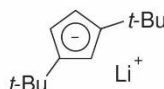
Li(acac)
Lithium acetylacetonate



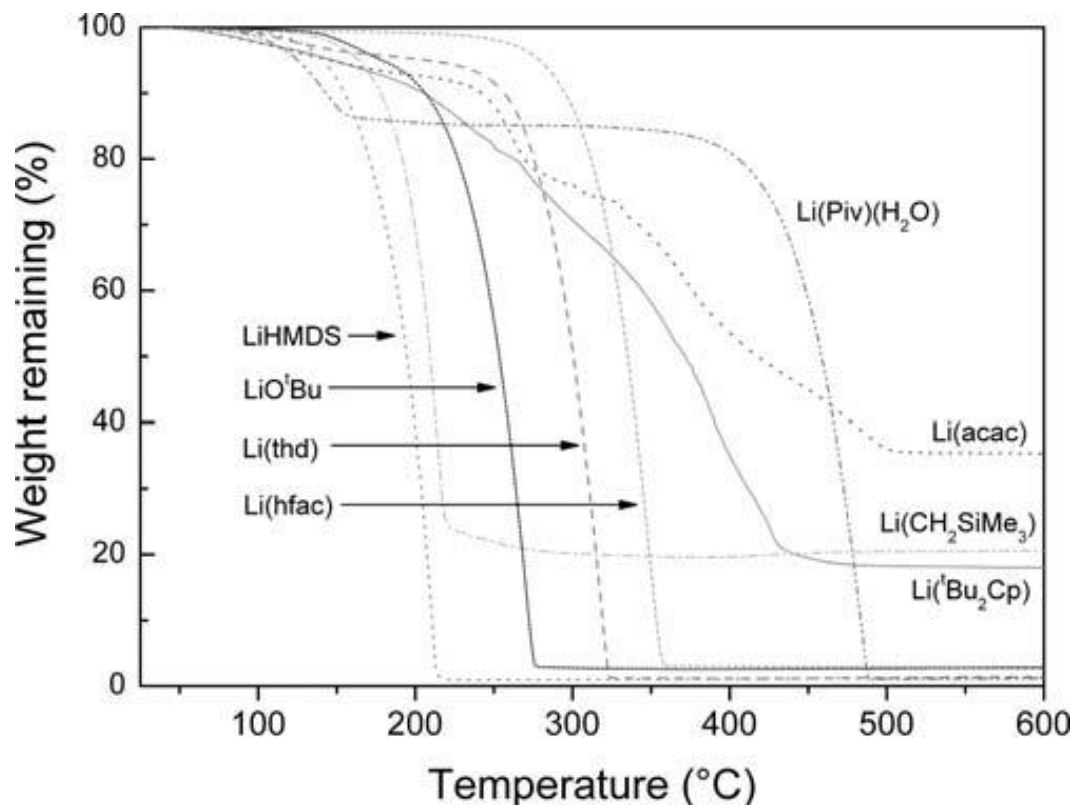
Li(Piv)(H₂O)
Lithium pivalate *H₂O



Li(CH₃SiMe₃)
Lithium trimethylsilane

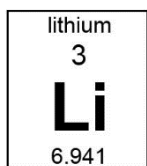


Li(^tBu2Cp)
Lithium Di-*tert*-butylcyclopentadiene

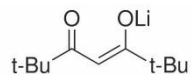


J. Hamalainen, J. Holopainen, F. Munnik, T. Hatanpaa, M. Heikkila, M. Ritala, and M. Leskela, *J. Electrochem. Soc.* **159**, A259 (2012).

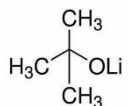
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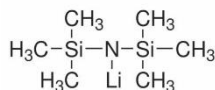
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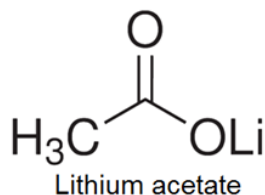
Li(thd)
Lithium (2,2,6,6-tetramethyl-3,5-heptanedionato)



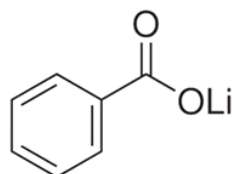
Li(O^tBu)
Lithium *tert*-butoxide



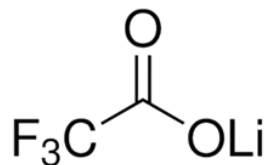
LiHMDS
Lithium hexamethyldisilazane
Lithium bis(trimethylsilyl)amide



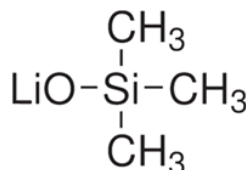
Lithium acetate



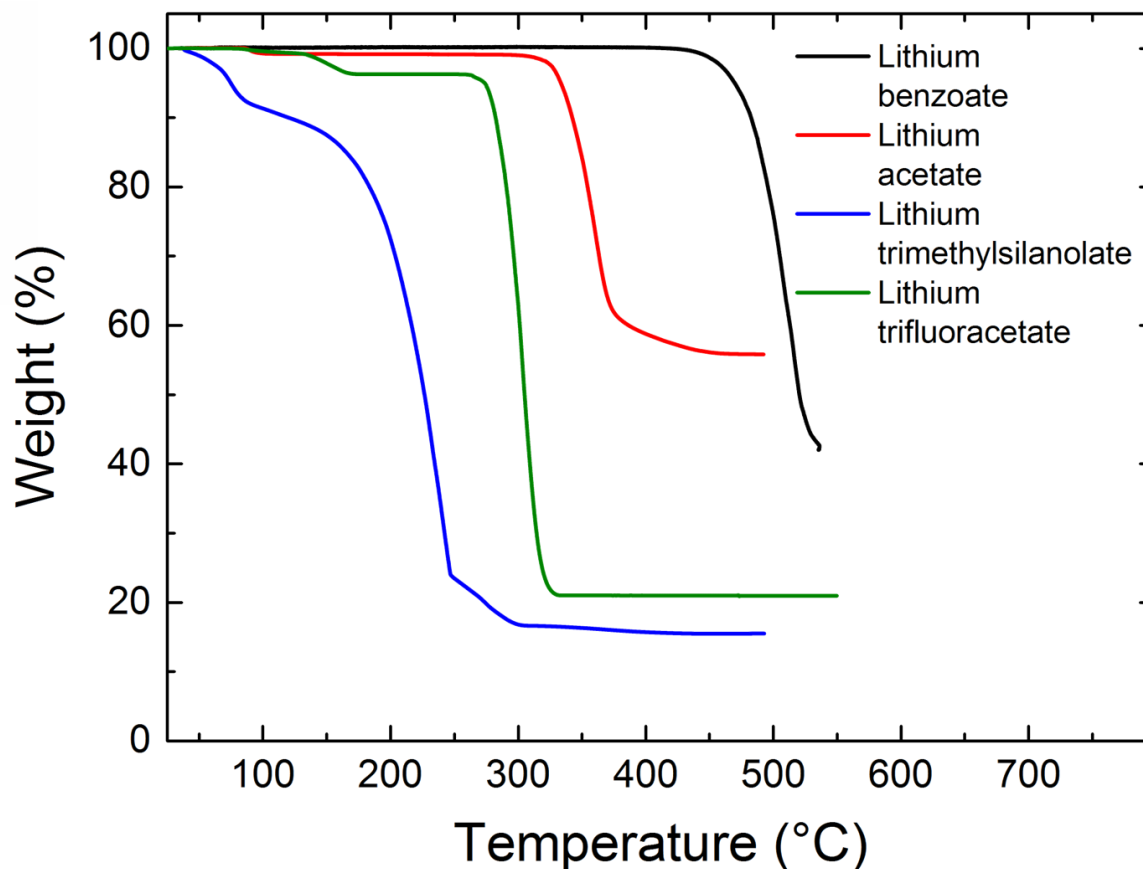
Lithium benzoate



Lithium trifluoroacetate

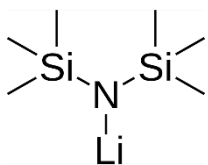


Lithium trimethylsilanolate

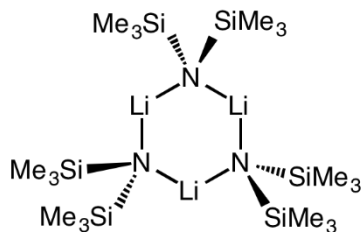


Why is not straight forward?

Monovalent, ...so are Cu(I)Cl, Ag⁺, In(I)Cl...
⇒ As monomer, what terminates the surface?

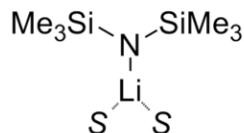


Does not exist

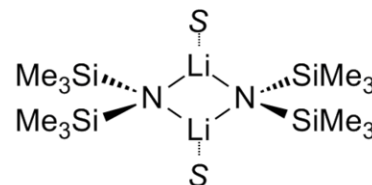


Sublimes as trimer

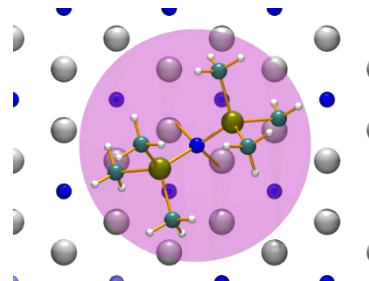
S = Solvent



Monomer and dimer in solvated form



Ref. to growth of Li₃N later...



Why is not straight forward?

Monovalent, ...so are Cu(I)Cl, Ag⁺, In(I)Cl...

⇒ As monomer, what terminates the surface?

Basic oxide?

⇒ Basic oxides tend to absorb water and CO₂...



Ref. to growth of Li₂CO₃ later...

Same for growth of Na and K compounds?

Yes...

Why is not straight forward?

Monovalent, ...so are Cu(I)Cl, Ag⁺, In(I)Cl...

⇒ As monomer, what terminates the surface?

Basic oxide?

⇒ Basic oxides tend to absorb water and CO₂...

Mobile ions?

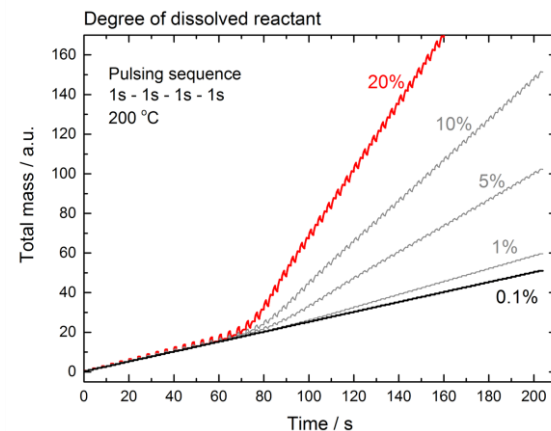
⇒ The aim is Li-ion conducting materials..., after all...



Ref. to growth of LiAlO₂ later...

⇒ Reservoir growth

Ref. to lithiation of MnO₂ and V₂O₅



Why is not straight forward?

Monovalent, ...so are Cu(I)Cl, Ag⁺, In(I)Cl...

⇒ As monomer, what terminates the surface?

Basic oxide?

⇒ Basic oxides tend to absorb water and CO₂...

Mobile ions?

⇒ The aim is Li-ion conducting materials..., after all...

Control composition?

⇒ A sum of the above...



Need a good selection of precursor chemistries

Why is not straight forward?

Monovalent, ...so are Cu(I)Cl, Ag⁺, In(I)Cl...

⇒ As monomer, what terminates the surface?

Basic oxide?

⇒ Basic oxides tend to absorb water and CO₂...

Mobile ions?

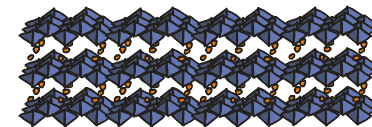
⇒ The aim is Li-ion conducting materials..., after all...

Control composition?

⇒ A sum of the above...

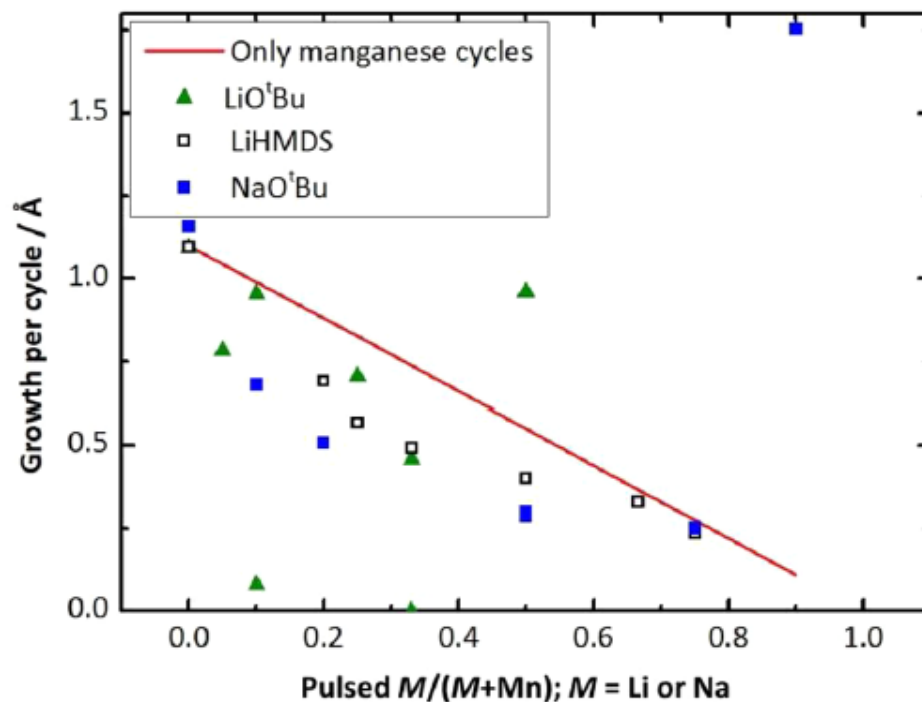
Freshness of precursor?

⇒ Batch differences...



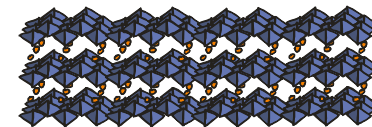
LiMn_2O_4 using $[\text{LiOtBu} + \text{H}_2\text{O}] + [\text{Mn}(\text{EtCp})_2 + \text{H}_2\text{O}]$

The same result with $[\text{Li}(\text{thd}) + \text{O}_3] + [\text{Mn}(\text{thd})_3 + \text{O}_3] \dots$



Almost no Li or Na incorporated into the film during growth

Figure 1. Film growth per cycle vs pulsing ratio (proportion of alkali-metal subcycles in a supercycle) using $\text{Mn}(\text{EtCp})_2$ as the Mn precursor. The line represents the theoretical growth per cycle with no contribution from alkali-metal subcycles to the film growth.



The same result with [Li(thd) + O₃] + [Mn(thd)₃ + O₃]...

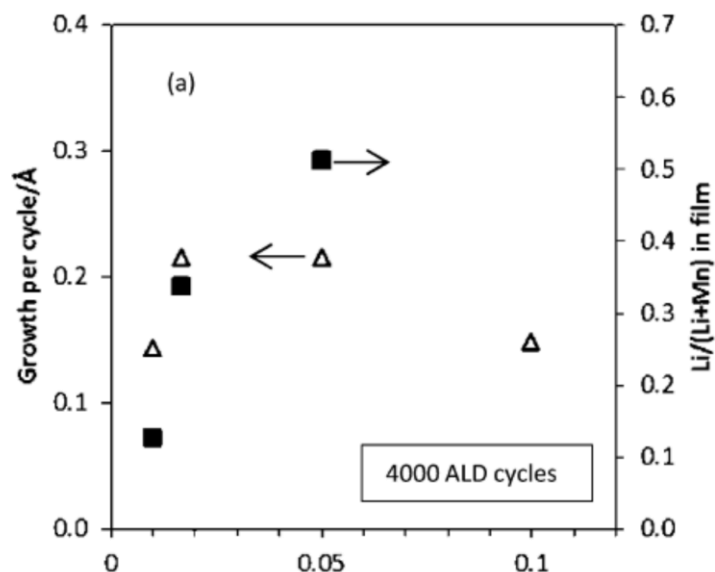


Figure 2. Growth per cycle and relative lithium composition for Li_xMn_yO_z films deposited with Li(thd) as the lithium precursor for films with (a) 4000 and (b) 1000 deposition cycles.

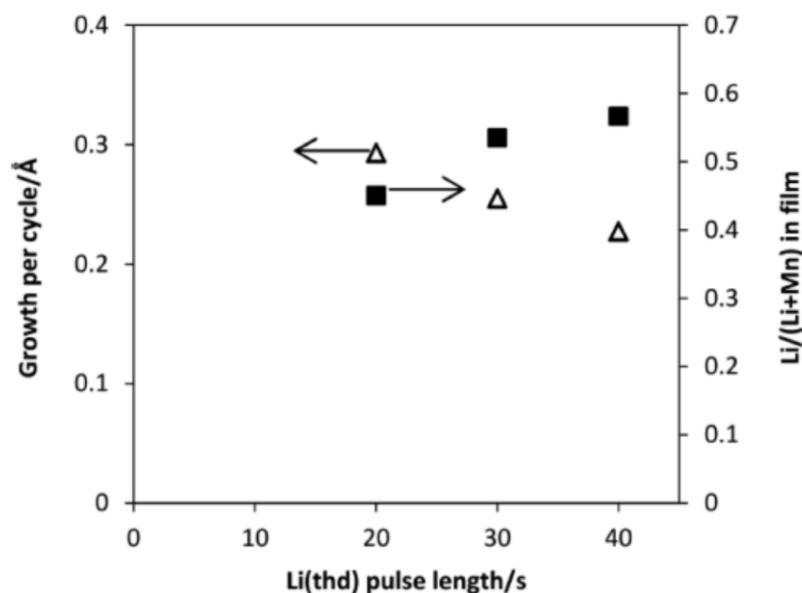
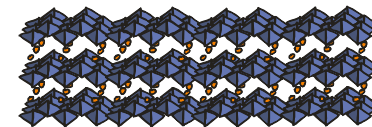


Figure 4. Film growth per cycle and relative lithium composition for Li_xMn₂O₄ films deposited with a pulsing sequence of 100 × {19 × [Mn(thd)₃ (1.5/1) + O₃ (5/5)] + [Li(thd) (X/10) + O₃ (5/5)]}.

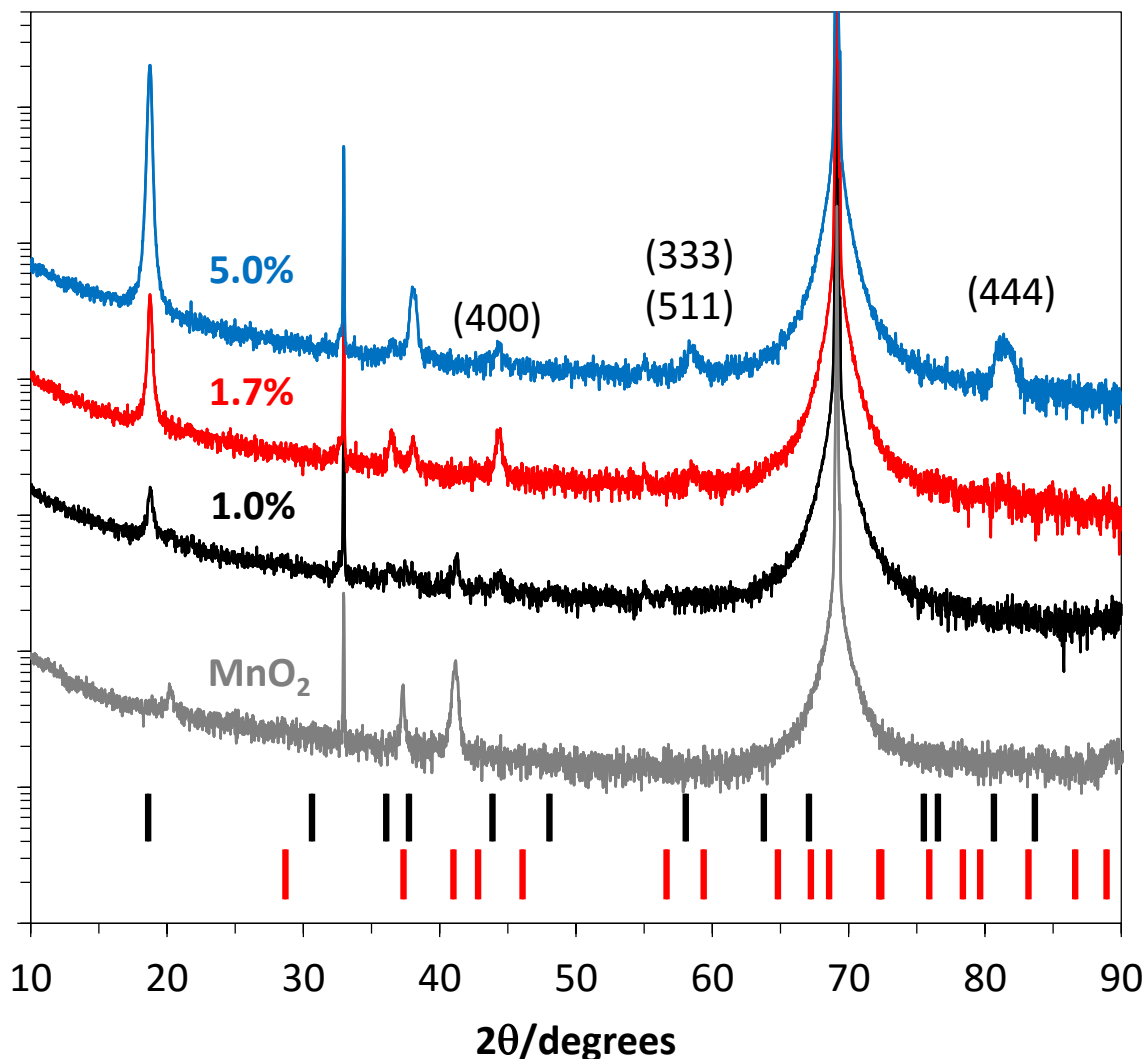
Saturation of Li content
for ca. 55%

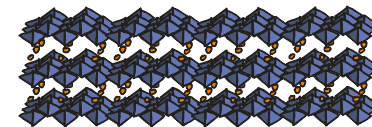


The same result with $[\text{Li}(\text{thd}) + \text{O}_3] + [\text{Mn}(\text{thd})_3 + \text{O}_3] \dots$

XRD for 4000-cycle films on Si(100):

- Spinel $\text{Li}_x\text{Mn}_2\text{O}_4$ | visible throughout the series
- MnO_2 impurity | with 1.0% pulsed Li
- Film with 5.0% pulsed Li [111] oriented





Gas phase lithiation

$[\text{Li}(\text{thd}) + \text{O}_3] = \text{fixed composition}$

$[\text{LiO}^t\text{Bu} + \text{H}_2\text{O}] = \text{fixed composition}$

...

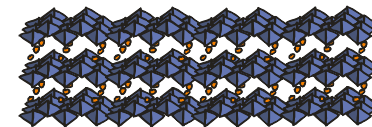
Grew films of:

V_2O_5 , MnO_2 , TiO_2 , Al_2O_3 , ZnO , Co_3O_4 , Fe_2O_3 , NiO

Tried lithiation using $[\text{Li}(\text{thd}) + \text{O}_3]$ or $[\text{LiO}^t\text{Bu} + \text{H}_2\text{O}]$

Got: unaltered films for TiO_2 , Al_2O_3 , ZnO , Co_3O_4 , Fe_2O_3 , NiO

-> V_2O_5 , MnO_2 ?



Gas phase lithiation: MnO_2

$[\text{Li}(\text{thd}) + \text{O}_3] = \text{fixed composition}$

$[\text{LiO}^t\text{Bu} + \text{H}_2\text{O}] = \text{fixed composition}$

...

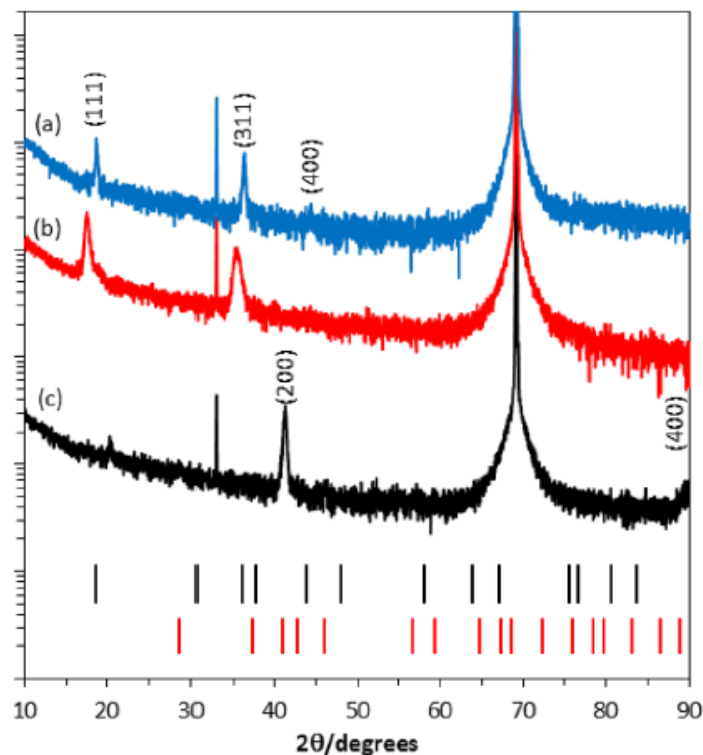
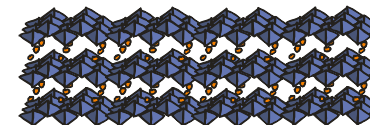


Figure 6. XRD diffractograms of (a) $\text{Li}(\text{thd})\text{-O}_3$ -treated (100 ALD cycles) and annealed MnO_2 , (b) $\text{Li}(\text{thd})\text{-O}_3$ -treated MnO_2 , and (c) as-deposited MnO_2 (110 nm). Films were deposited on $\text{Si}(100)$. Black and red tick marks below the graphs refer to LiMn_2O_4 spinel (35-0782) and pyrolusite $\beta\text{-MnO}_2$ (24-0735), respectively.

As lithiated and annealed: LiMn_2O_4

As lithiated: LiMn_2O_4

Started with: MnO_2



Gas phase lithiation: MnO_2

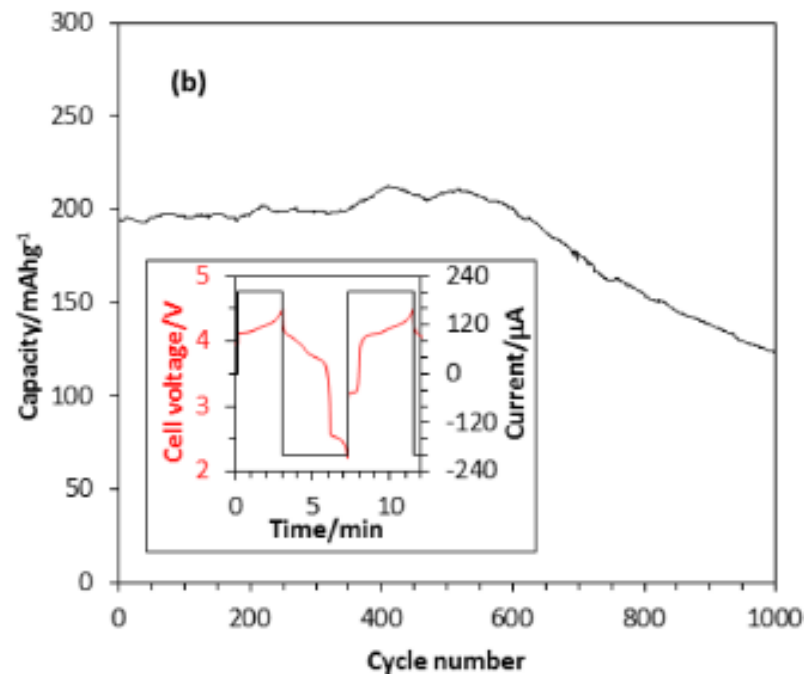
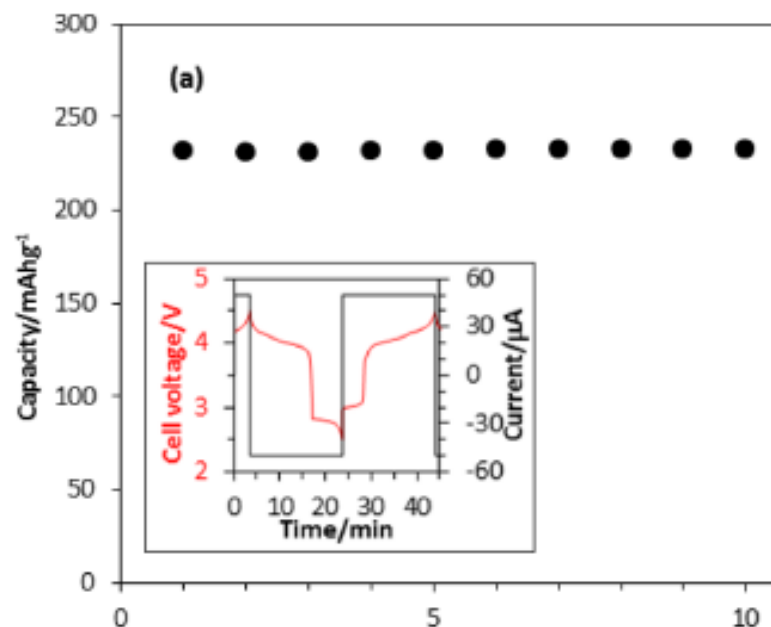
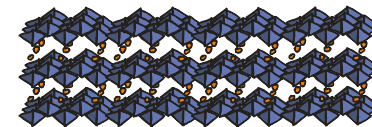


Figure 11. Discharge capacities and first-cycle potentiograms (insets) of an 86-nm MnO_2 film treated with 200 cycles of $\text{LiO}^t\text{Bu} + \text{H}_2\text{O}$: (a) 10 charge–discharge cycles with 50 μA , (b) 1000 charge–discharge cycles with 200 μA .



Gas phase lithiation: V_2O_5

[Li(thd) + O_3] = fixed composition

[LiO^tBu + H_2O] = fixed composition

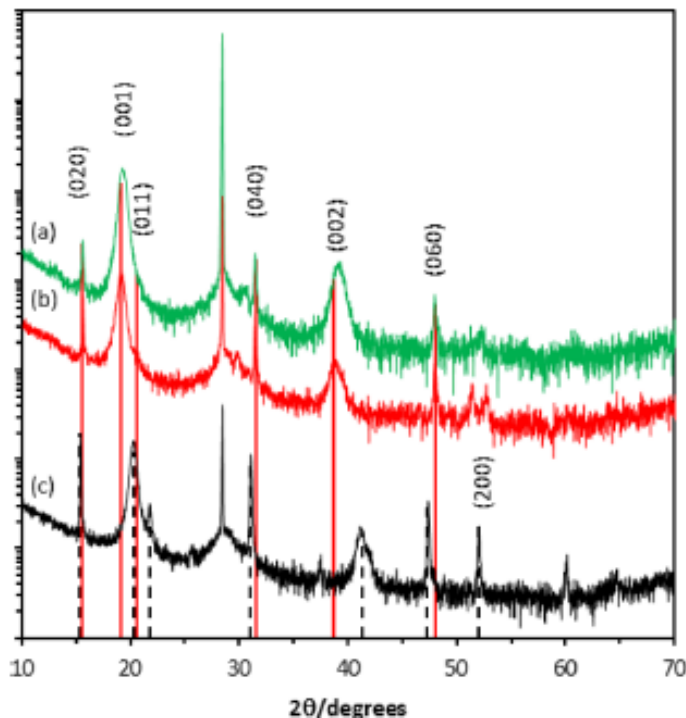


Figure 8. XRD diffractograms of (a) $Li_xV_2O_5$ films made with LiO^tBu-H_2O treatment, (b) $Li_xV_2O_5$ films made with $Li(thd)-O_3$ treatment, and (c) parent V_2O_5 film on Si(111). Indexing refers to the $Pmmn$ space group for V_2O_5 and $\epsilon-Li_xV_2O_5$,³⁵ with unit cell dimensions of $a = 3.57 \text{ \AA}$, $b = 11.54 \text{ \AA}$, $c = 4.38 \text{ \AA}$ and $a = 3.57 \text{ \AA}$, $b = 11.36 \text{ \AA}$, $c = 4.65 \text{ \AA}$, respectively. Dashed black lines and red lines refer to $\alpha-V_2O_5$ and $\epsilon-Li_xV_2O_5$, respectively.

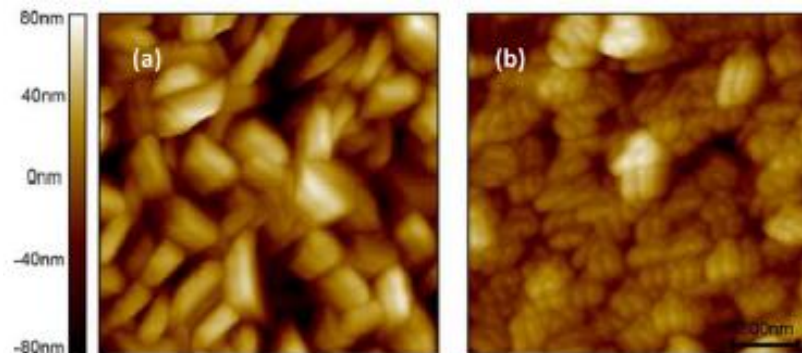
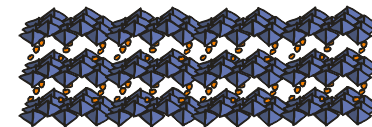


Figure 9. AFM images of (a) V_2O_5 film and (b) V_2O_5 film treated with 100 cycles of $LiO^tBu + H_2O$.

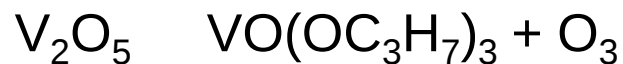
As LiO^tBu lithiated: $\epsilon-Li_xV_2O_5$

As $Li(thd)$ lithiated: $\epsilon-Li_xV_2O_5$

Started with: $\alpha-V_2O_5$



J.C. Badot, ..., D. Lincotb, *Electrochem. Solid-State Lett.* **3** (2000) 485.



X. Chen, ..., G. Rubloff, *Chem.Mater.* **24** (2012) 1255.

O_3 -based ALD of crystalline V_2O_5 cathodes

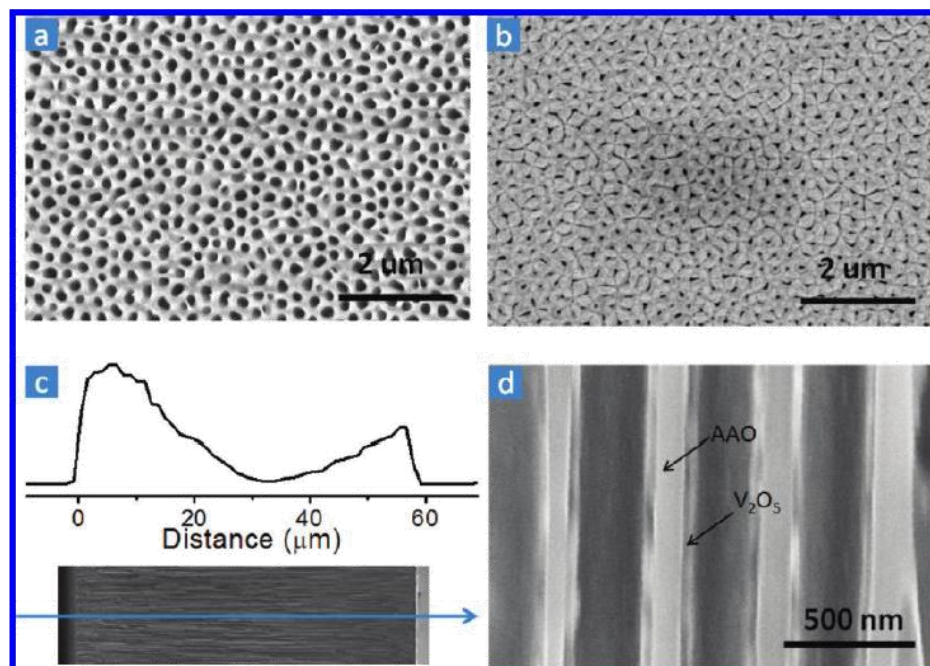
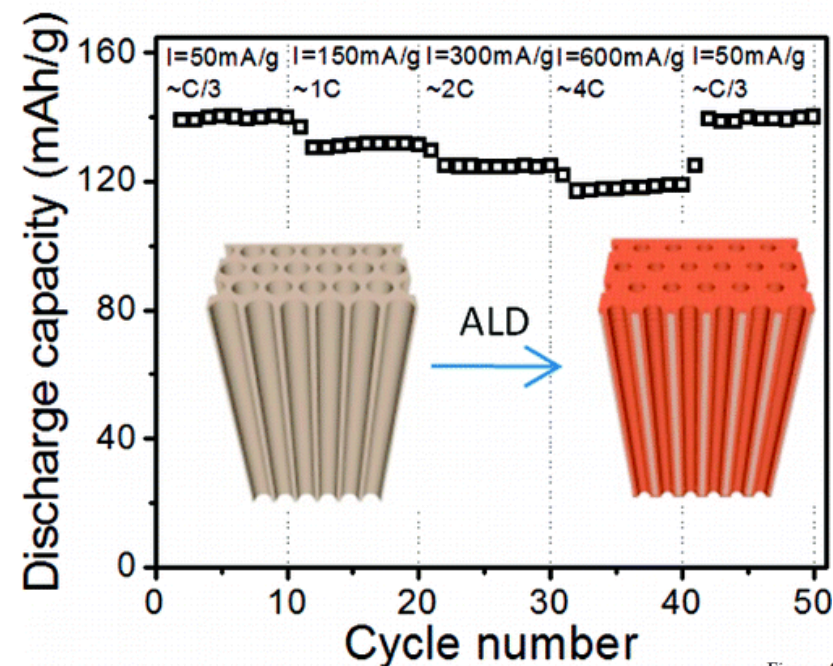
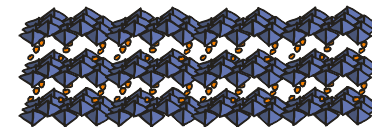
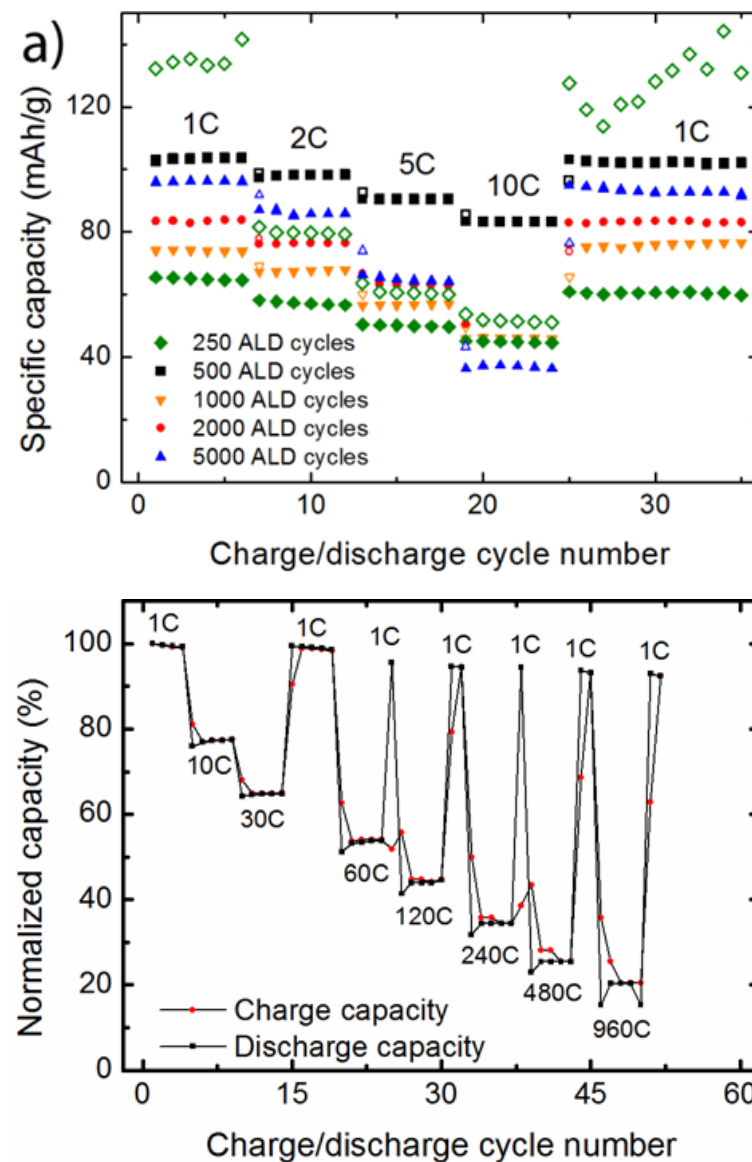
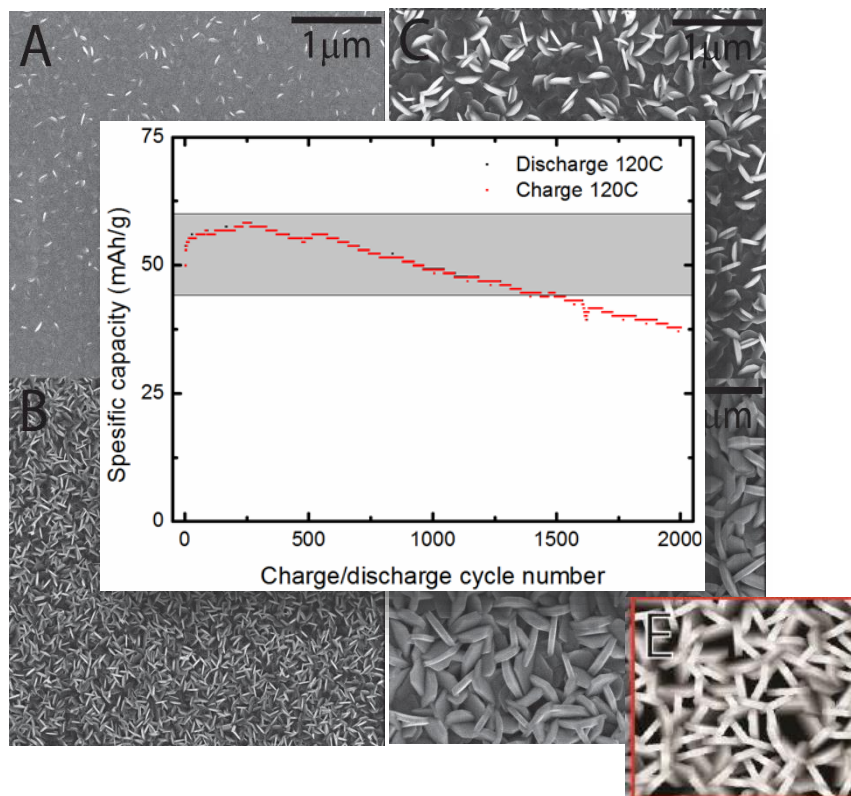


Figure 4. SEM of the AAO template (a) before and (b) after 1000 cycle ozone-based ALD V_2O_5 film deposition; (c) EDX line scan of V signal through the cross-section of V_2O_5 coated AAO template; (d) SEM image of V_2O_5 nanotubes inside AAO pores.

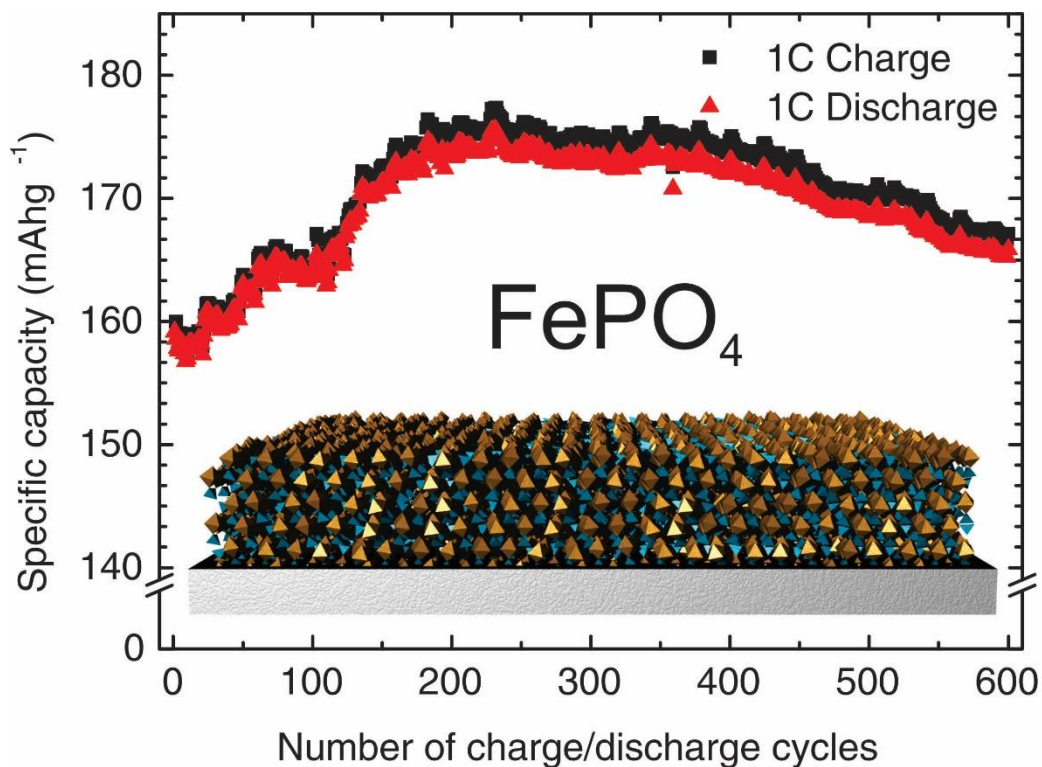
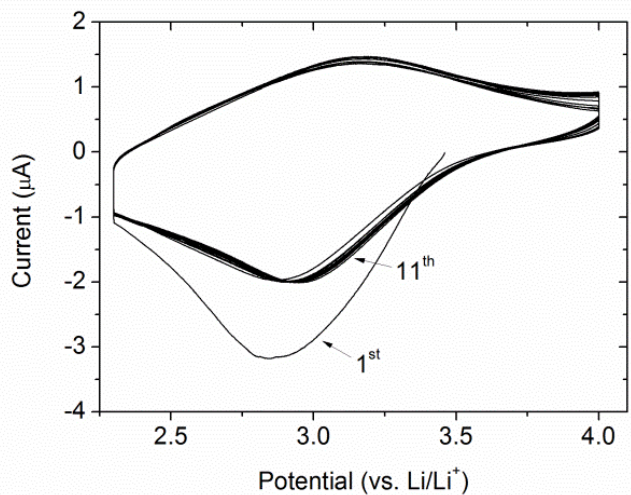
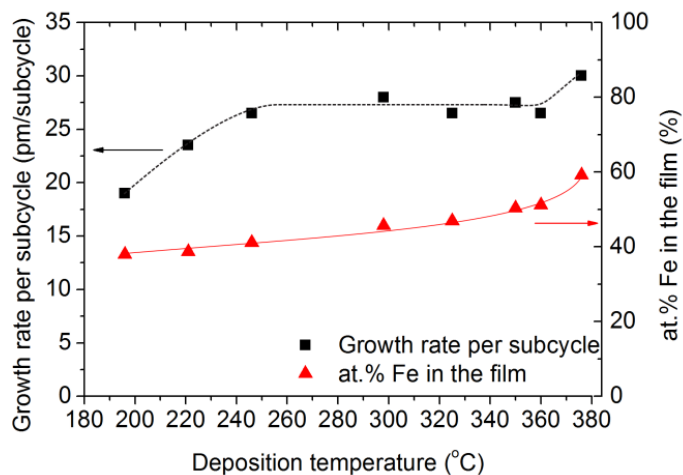
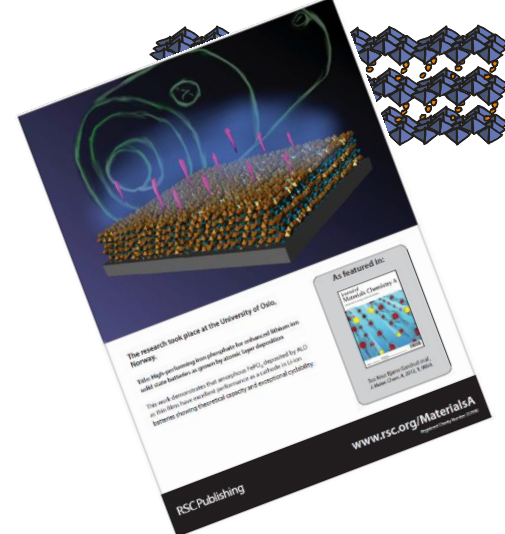


E. Østreng, ..., H. Fjellvåg,
J. Mater. Chem. A, **2** (2014) 15044

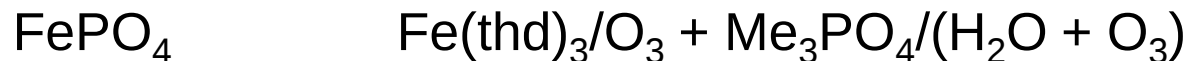


FePO₄

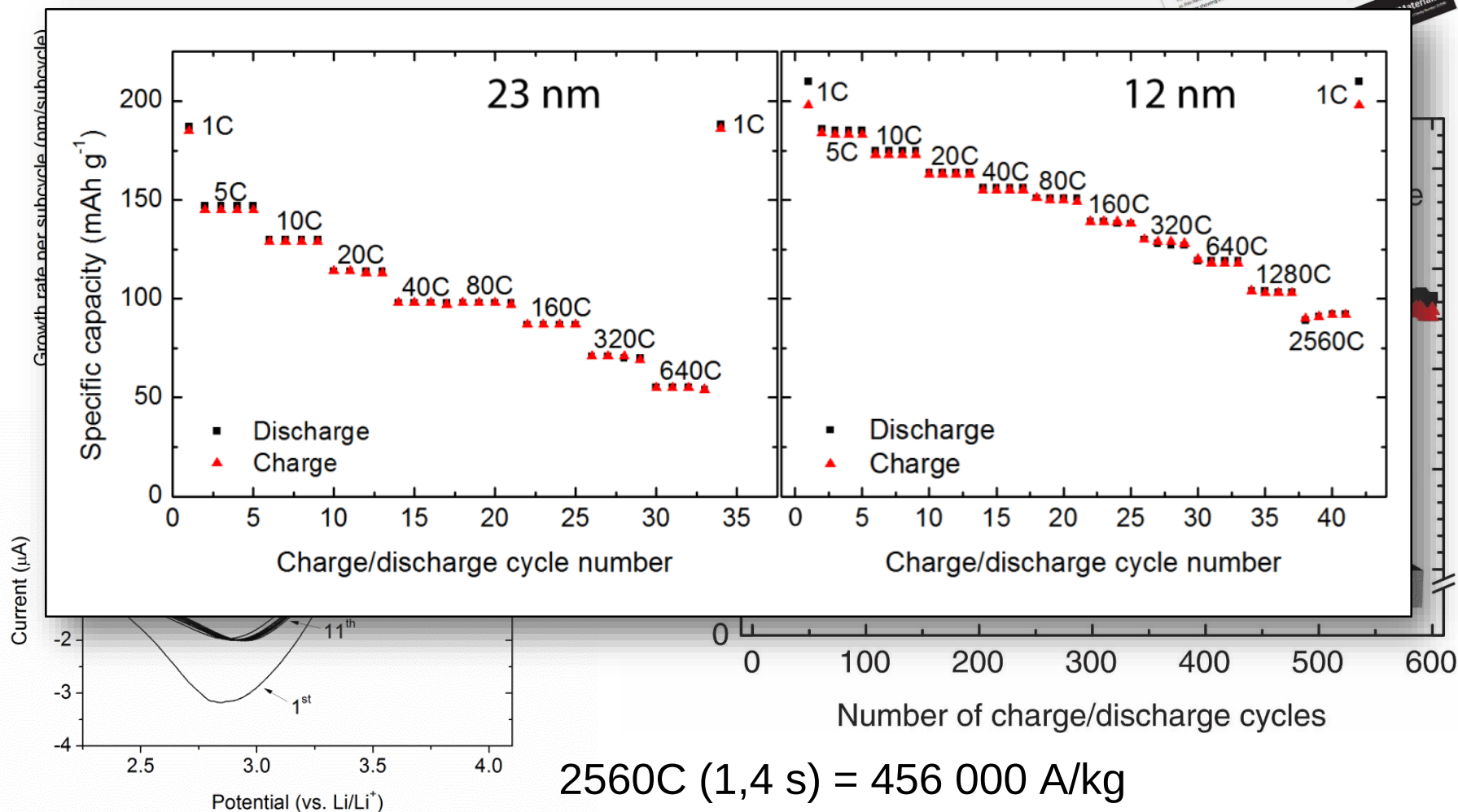
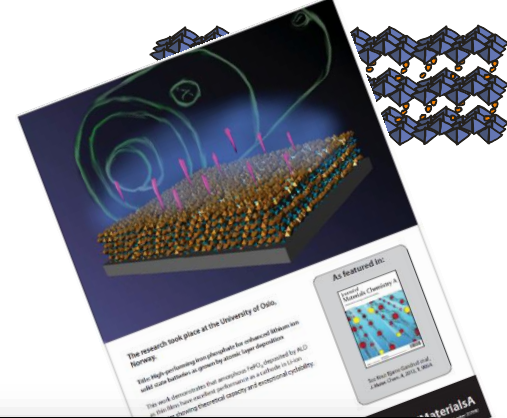
FePO₄ Fe(thd)₃/O₃ + Me₃PO₄/(H₂O + O₃)
K.B. Gandrud, ..., H. Fjellvåg, *J. Mater. Chem A*. **1** (2013) 9054



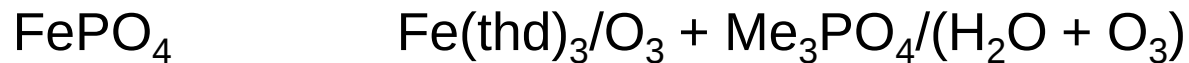
FePO₄



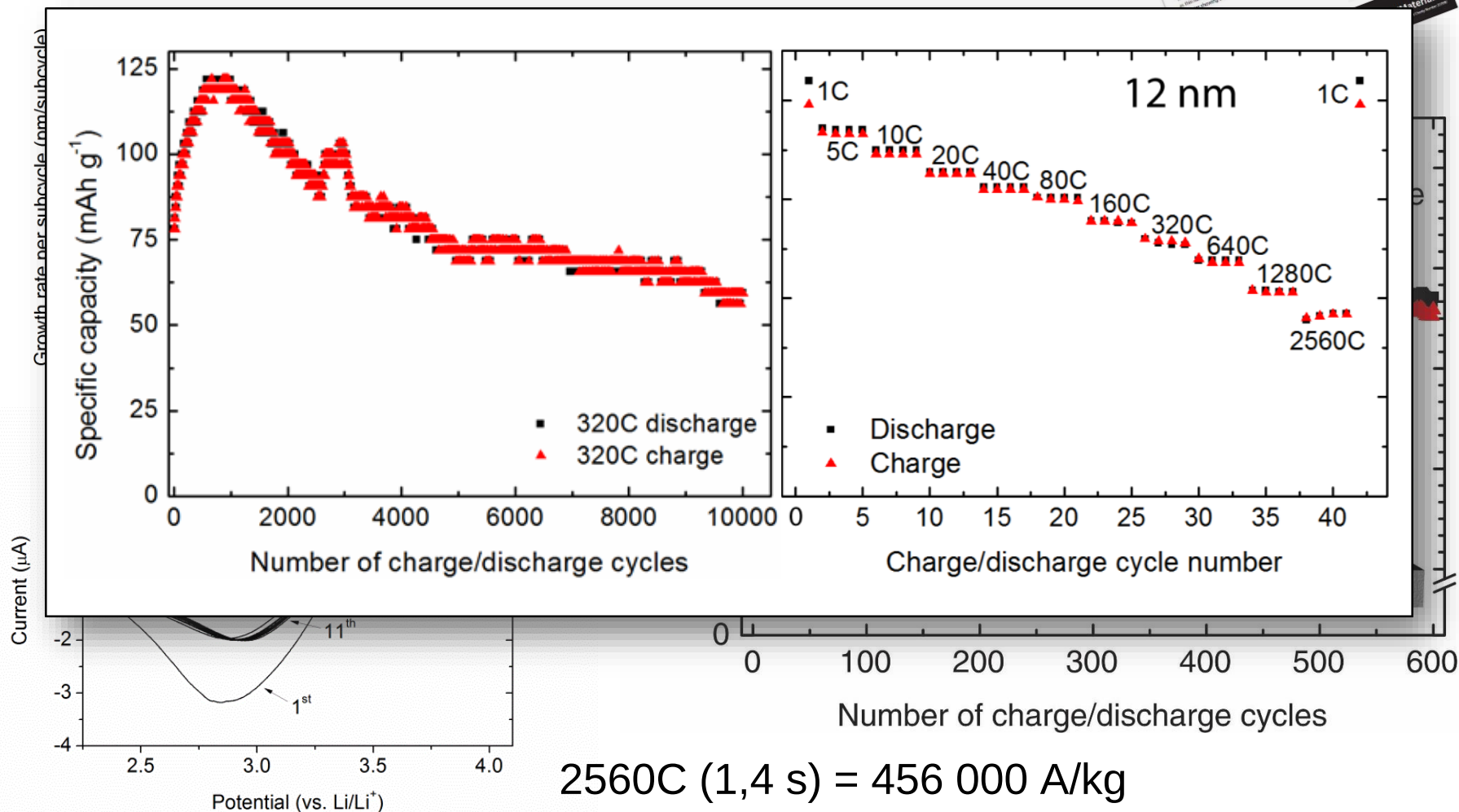
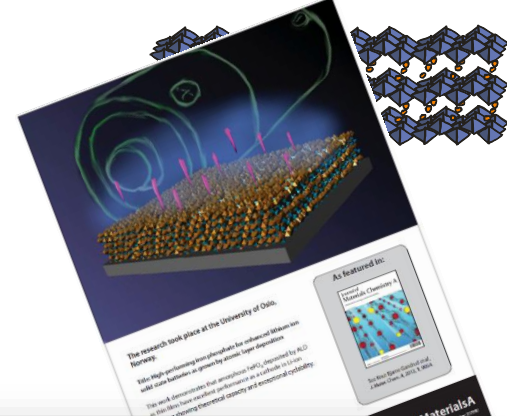
K.B. Gandrud, ..., H. Fjellvåg, *J. Mater. Chem A*. **1** (2013) 9054



FePO₄

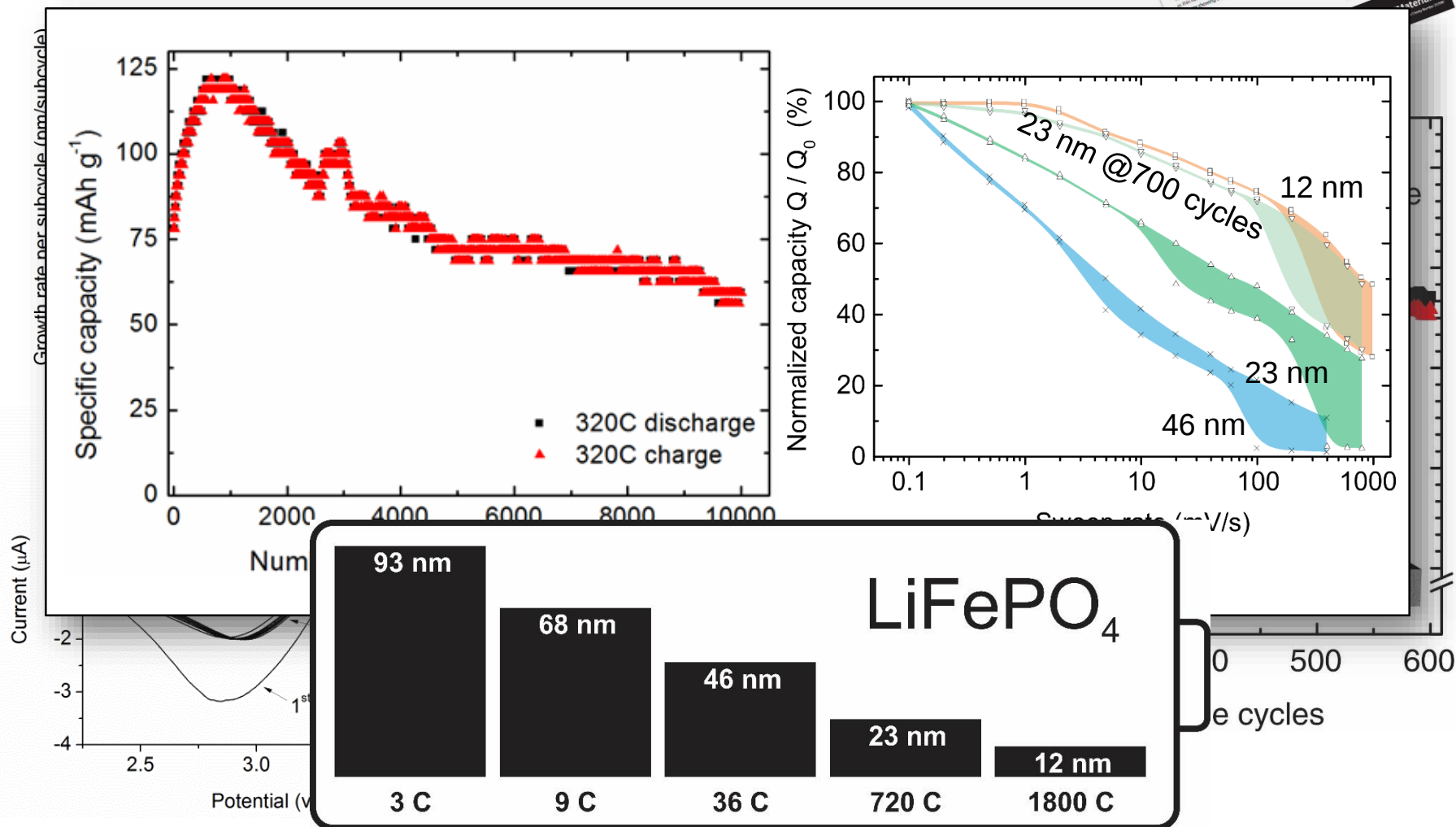
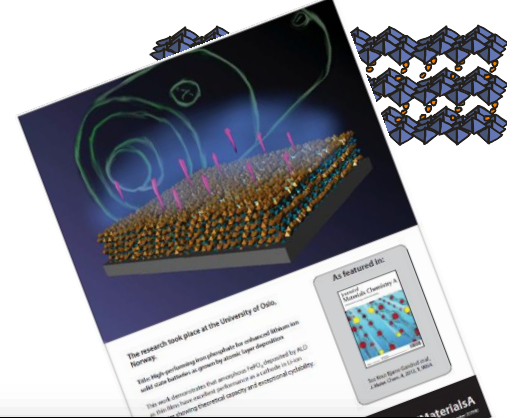


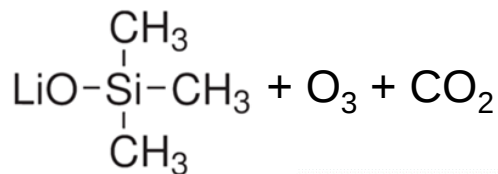
K.B. Gandrud, ..., H. Fjellvåg, *J. Mater. Chem A*. **1** (2013) 9054



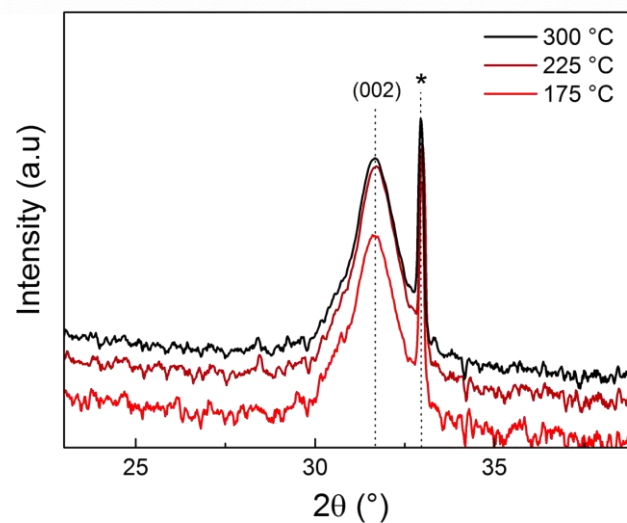
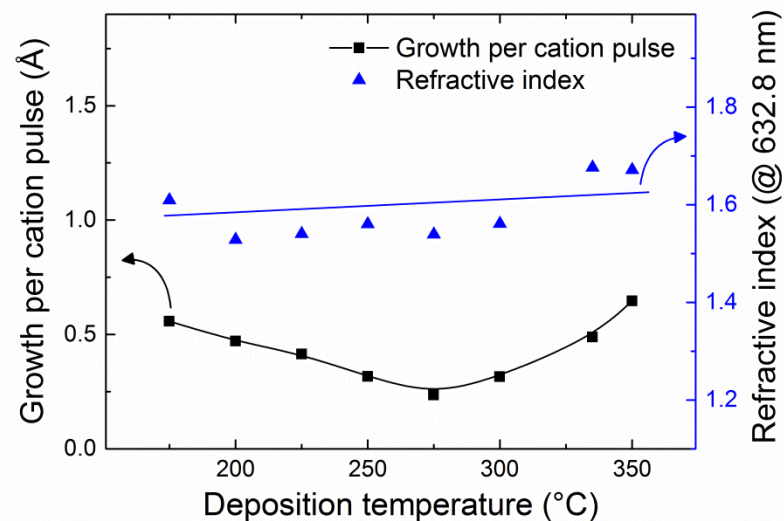
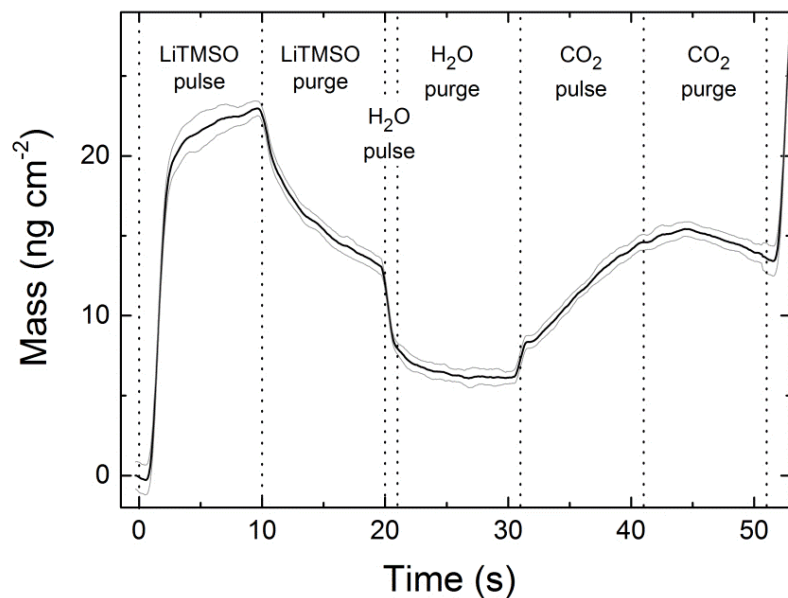
FePO₄

FePO₄ Fe(thd)₃/O₃ + Me₃PO₄/(H₂O + O₃)
K.B. Gandrud, ..., H. Fjellvåg, *J. Mater. Chem A*. **1** (2013) 9054





Lithium
trimethylsilanolate



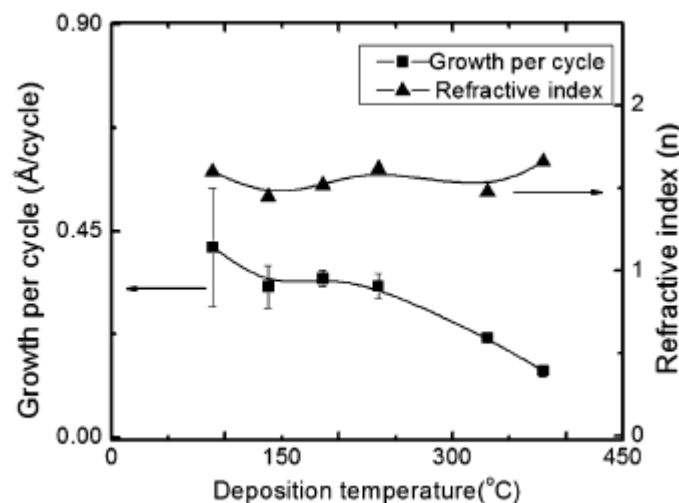
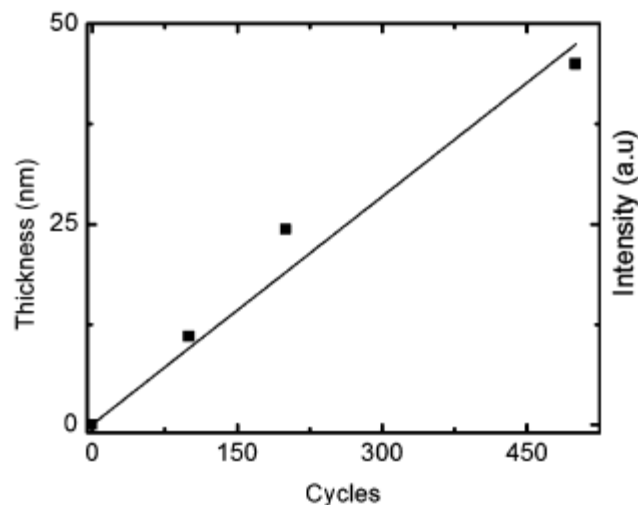
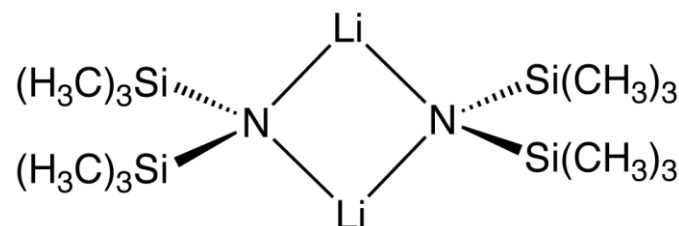


LiHMDS (Li(OH), Li₂CO₃, Li₃N)

[LiHMDS + H₂O] = reacts fast with air

[LiHMDS + NH₃] = reacts faster with air
- capped with MoN_x

[LiHMDS + H₂O + CO₂] = Li₂CO₃



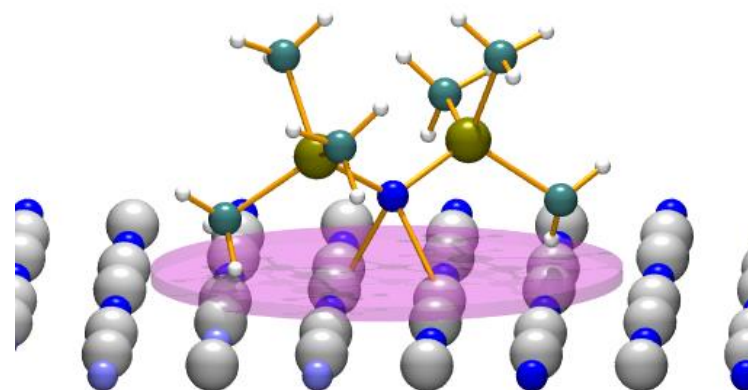
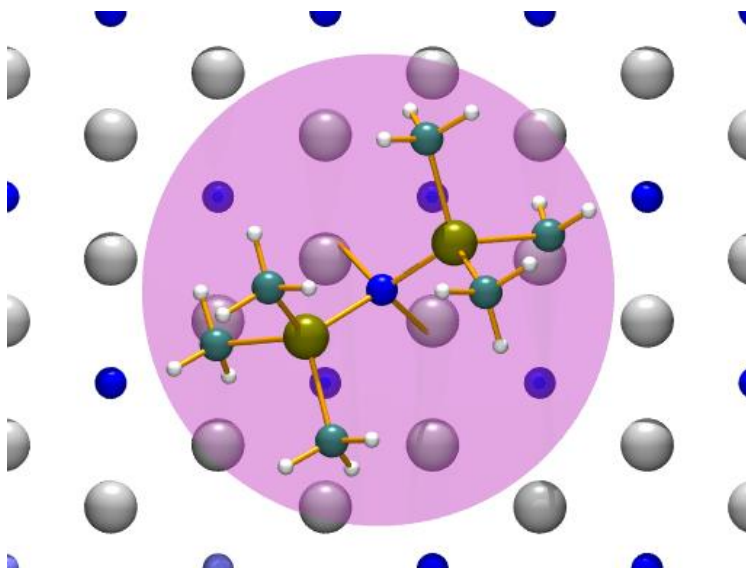
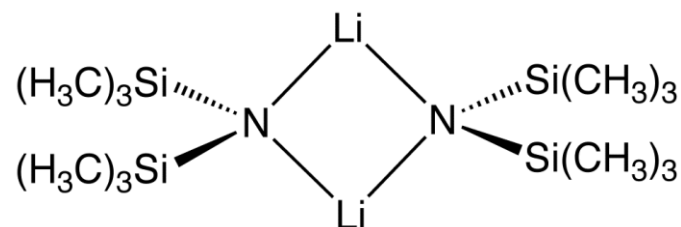


LiHMDS (Li(OH), Li₂CO₃, Li₃N)

[LiHMDS + H₂O] = reacts fast with air

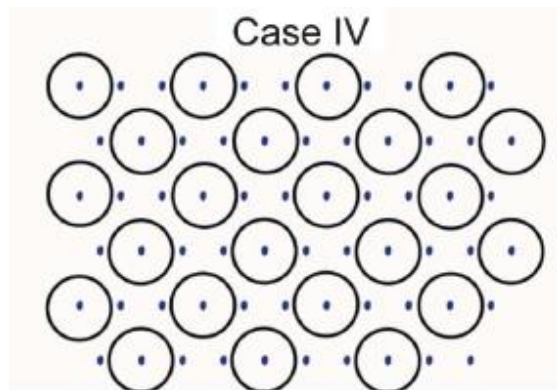
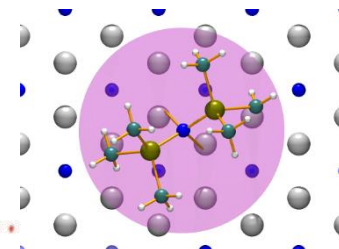
[LiHMDS + NH₃] = reacts faster with air
- capped with MoN_x

[LiHMDS + H₂O + CO₂] = Li₂CO₃





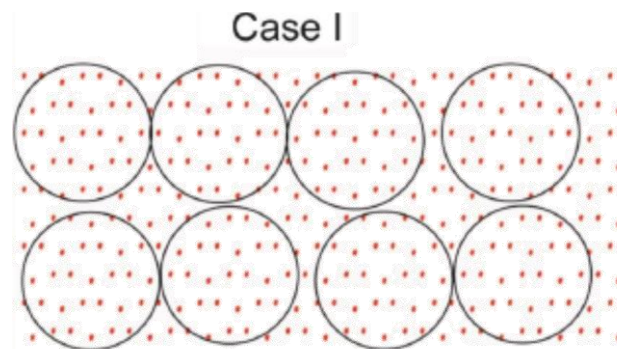
LiHMDS (Li(OH), Li₂CO₃, Li₃N)



GPC (Li₃N) = 0.73 Å/cycle

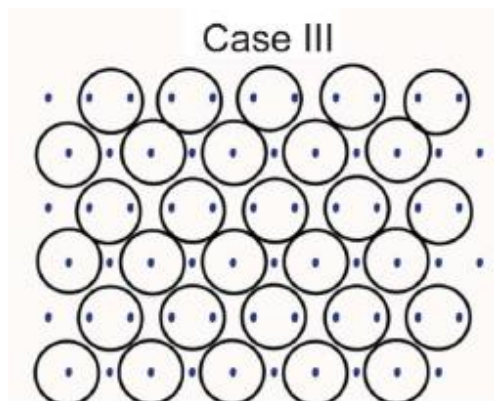
• N

• O



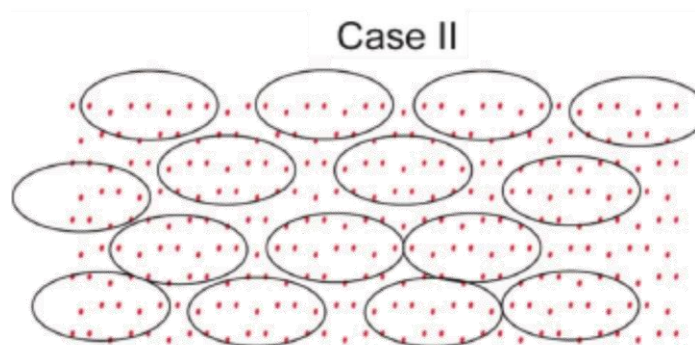
GPC (Li₂CO₃) = 0.23 Å/cycle

GPC@331 °C = 0.23 Å/cycle



GPC (Li₃N) = 1.1 Å/cycle

GPC@167 °C = 1.1 Å/cycle



GPC (Li₂CO₃) = 0.42 Å/cycle

GPC@89 °C = 0.41 Å/cycle



LiHMDS (Li-Si-O by [LiHMDS + O₃])

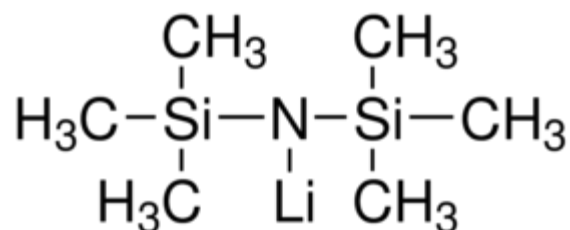


FIG. 7. (Color online) Photographs of the soda lime glass substrate after ALD lithium silicate film deposition at 300 °C.

TABLE I. Elemental compositions of the lithium silicate thin films as measured with ERDA.

	Deposition temperature					
	150 °C	200 °C	250 °C	300 °C	350 °C	400 °C
Li (at. %)	31.8	29.8	32.5	30.8	30.0	37.5
Si (at. %)	11.2	13.9	16.1	17.7	18.5	15.1
O (at. %)	44.2	48.7	46.3	48.6	49.6	45.9
C (at. %)	3.94	0.96	0.65	0.32	0.14	0.15
H (at. %)	8.78	6.63	4.60	2.70	1.71	1.29
Thickness (nm)	62	62	86	120	140	172
Density (g/cm ³)	2.48	2.26	2.32	2.36	2.39	2.5
Li:Si:O ratio	2.8:1:4.0	2.1:1:3.5	2.0:1:2.9	1.7:1:2.8	1.6:1:2.7	2.5:1:3.0

Li-content decreased with increasing temperature

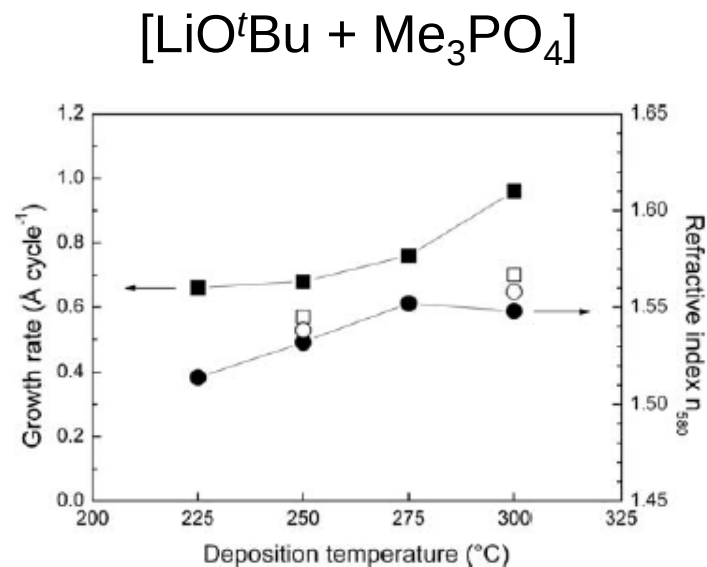
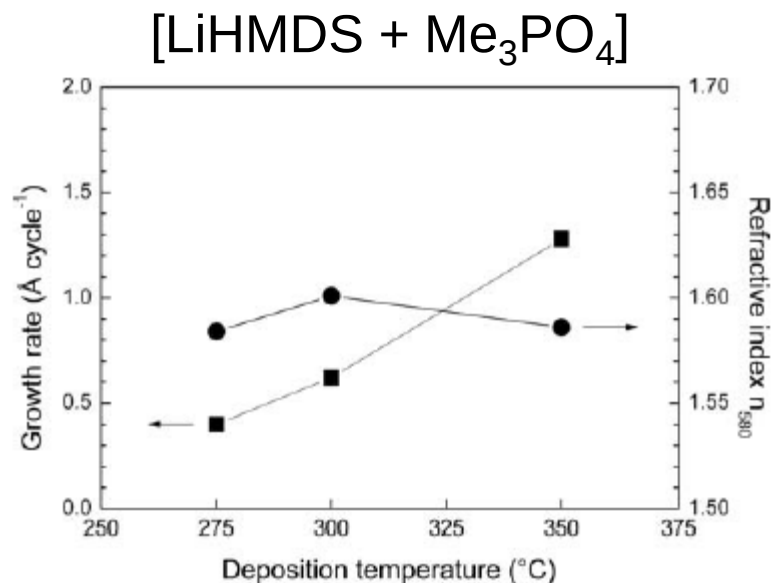


Figure 2. Growth rates and refractive indexes of lithium phosphate films as a function of deposition temperature. The precursor pulses and purges were either 2 s (open symbol) or 4 s (solid) each. A total of 1000 cycles were applied for each deposition.

J. Hämäläinen, ... M. Leskelä, *J. Electrochem. Soc.* **159** (2012) A259.



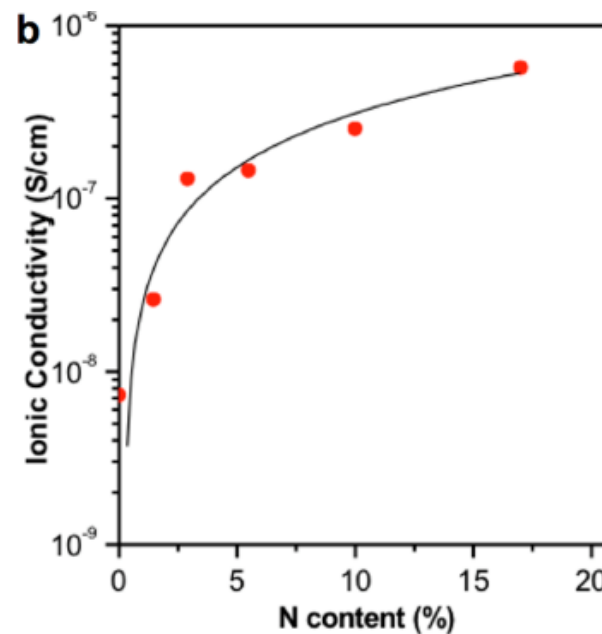
LiPON

$[\text{LiO}^t\text{Bu} + \text{H}_2\text{O} + \text{TMP} + {}^p\text{N}_2]$

LiPON (250 °C), 1.05 Å/s,

N content controlled by ${}^p\text{N}_2$ pulse length.

A. C. Kozen, A. J. Pearce, C.-F. Lin, M. Noked G. W. Rubloff, *Chemistry of Materials*, **2015**, 27, 5324-5331.

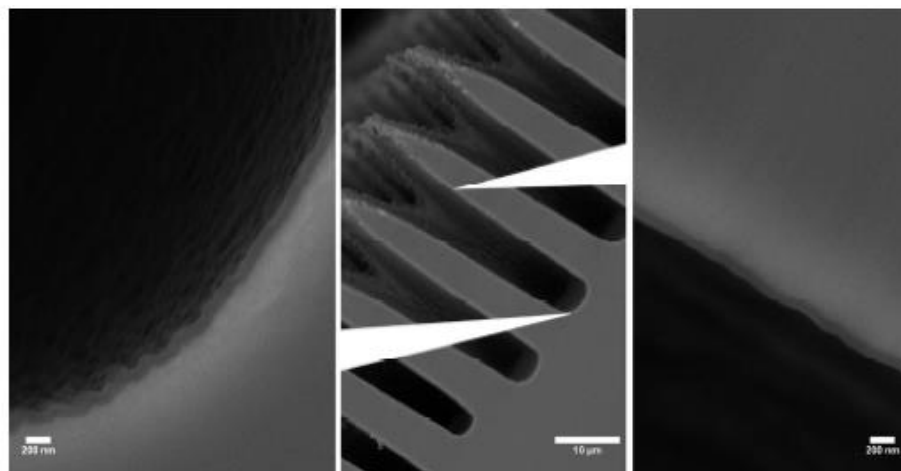


$[(\text{H}_2\text{NP}(\text{O})(\text{OC}_2\text{H}_5)_2) + \text{LiHMDS}]$

LiPON (250 – 330 °C), 0.7 – 1.0 Å/s.

Ionic conductivity of
 $6.6 \times 10^{-7} \text{ Scm}^{-1}$ @RT.

M. Nisula, Y. Shindo, H. Koga M. Karppinen, *Chemistry of Materials*, **2015**, 27, 6987-6993.





LiAlO₂

[LiO^tBu + H₂O] + [TMA + H₂O] @ 225 °C

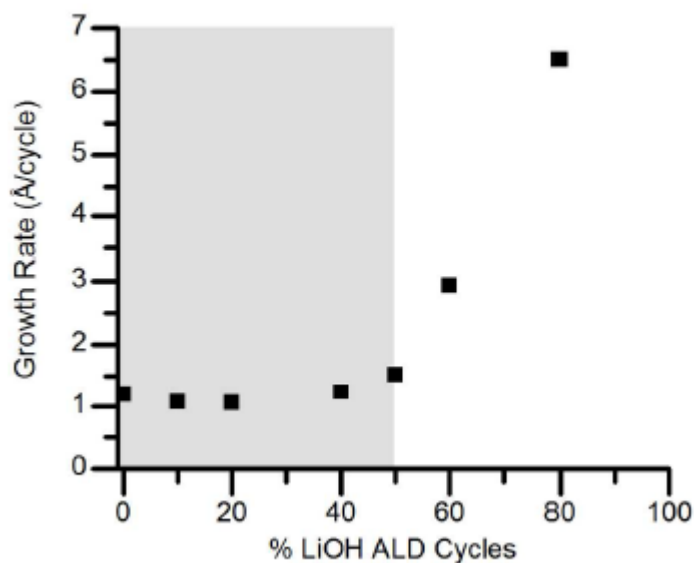


Figure 7. LiAlO_x growth rate measured by ellipsometry as a function of % LiOH ALD cycles. Gray shaded area designates the region of stable growth with constant, linear growth as a function of ALD cycles.

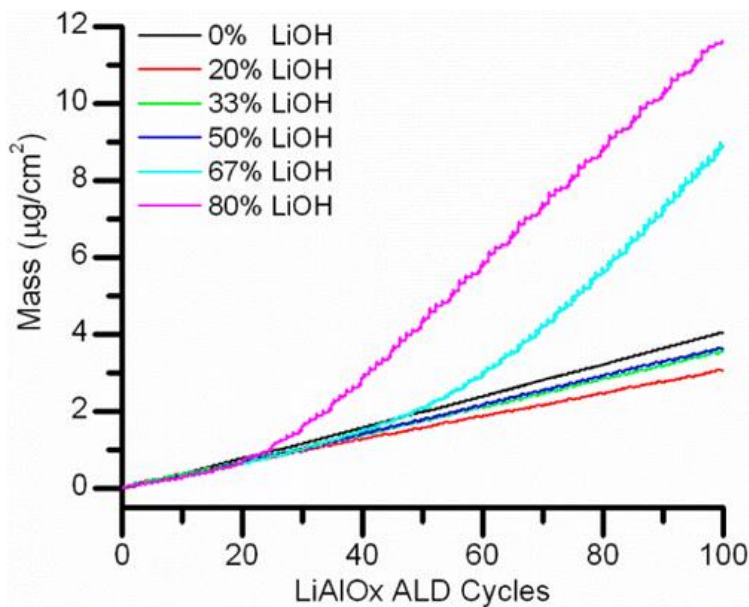


Figure 4. QCM mass gain profiles acquired over 100 cycles of LiAlO_x ALD using 0%, 20%, 33%, 50%, 67%, and 80% LiOH cycles.

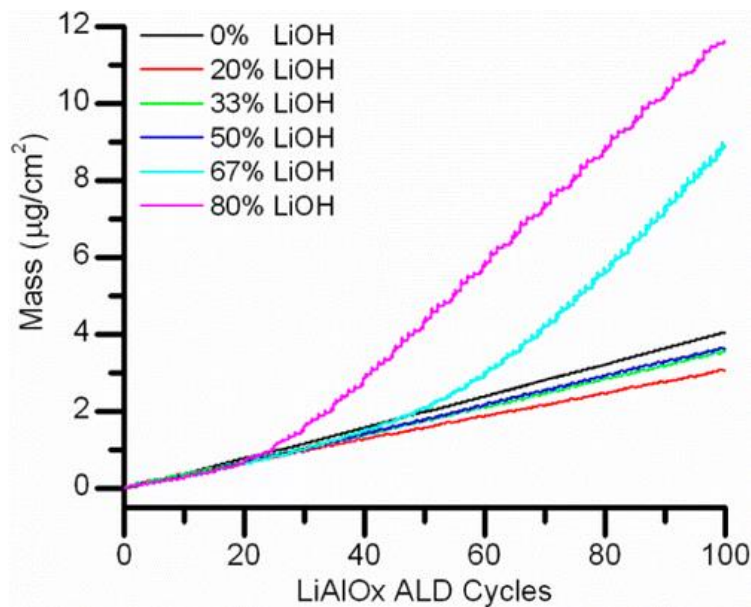
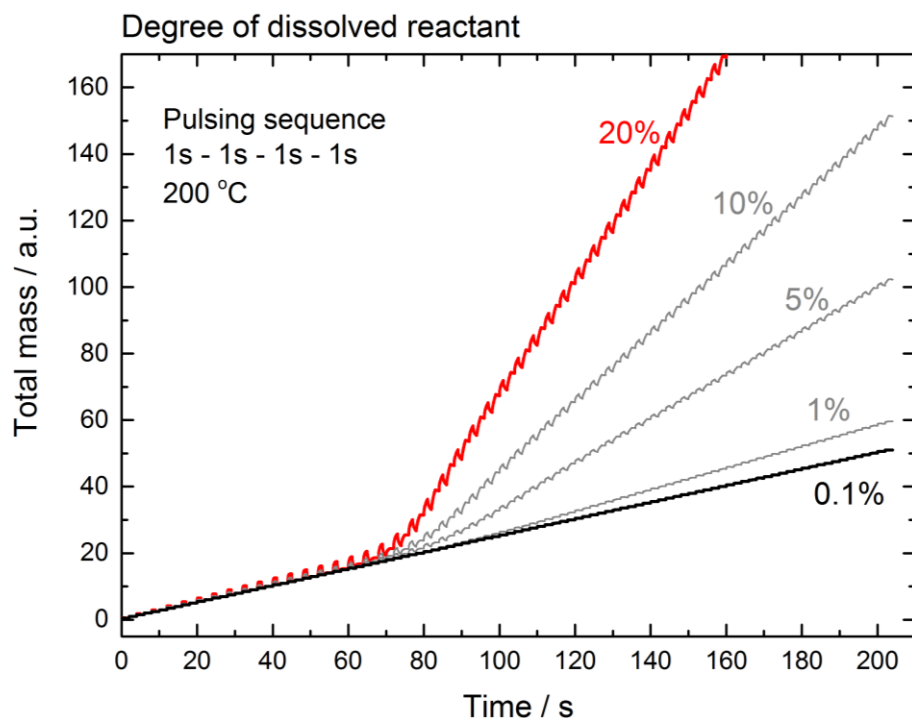
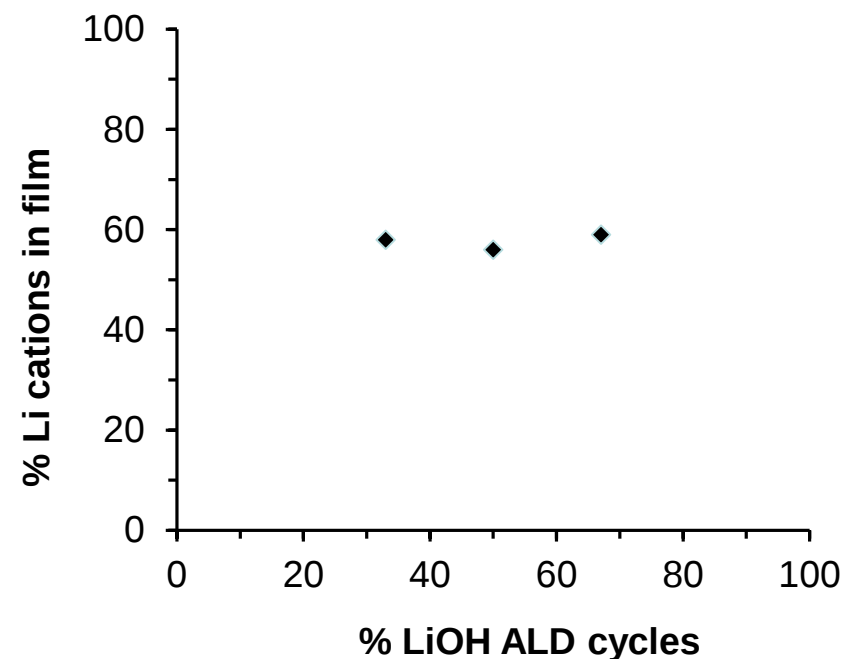
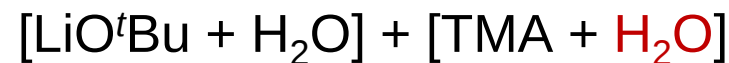


Figure 4. QCM mass gain profiles acquired over 100 cycles of LiAlO_x ALD using 0%, 20%, 33%, 50%, 67%, and 80% LiOH cycles.

O. Nilsen, The reservoir effect and its implications, *AVS ALD2015*.

D.J. Comstock, and J. W. Elam, *J. Phys. Chem. C.* **117** (2013) 1677.



V. Miikkulainen, ... H. Fjellvåg, *J. Vac. Sci. Technol. A* **33** (2015) 01A101

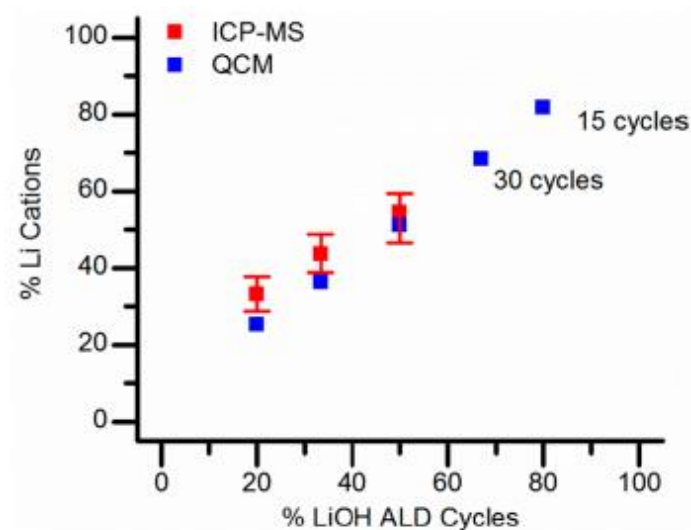
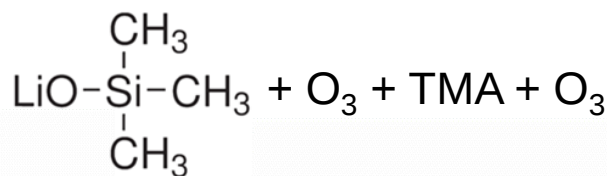
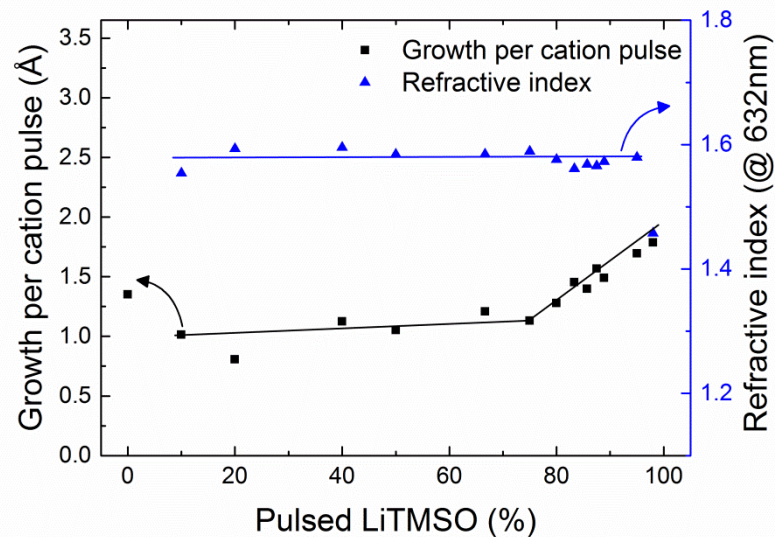
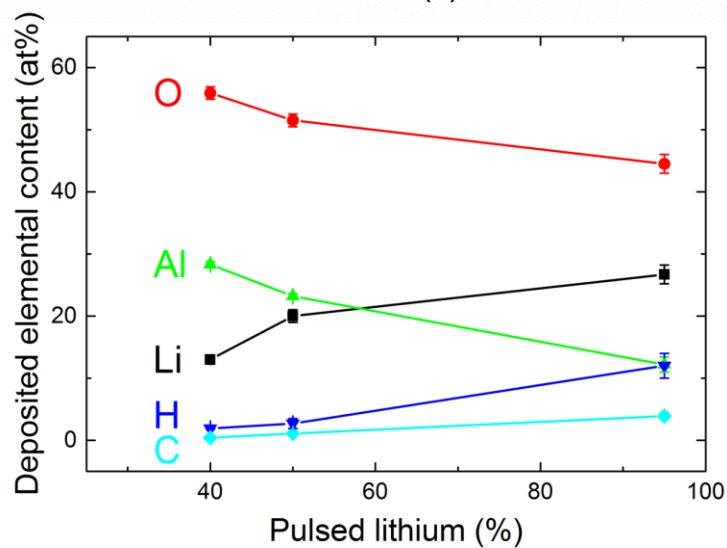
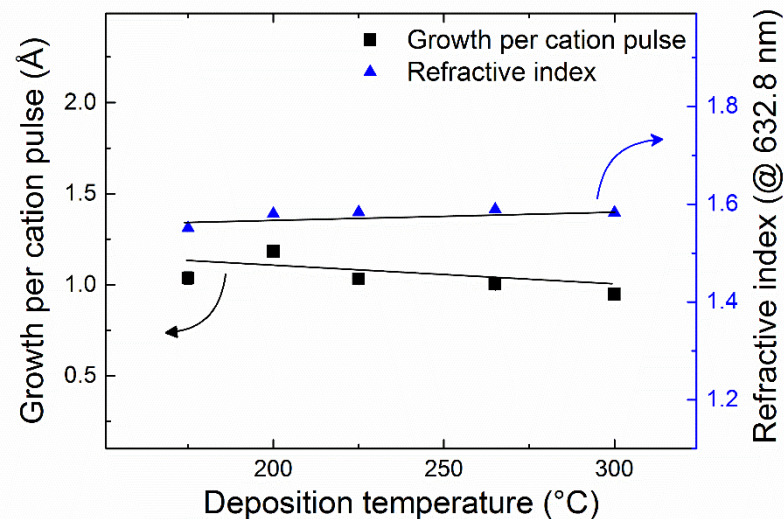
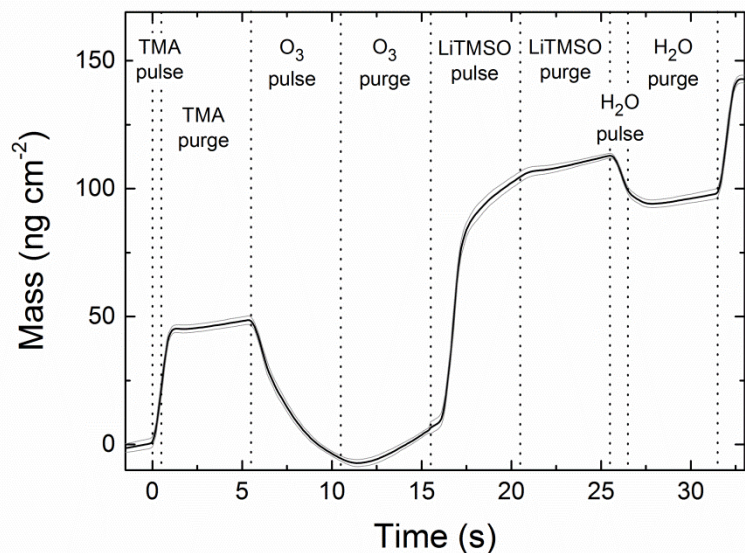


Figure 8. Li cation percentage in LiAlO_x films measured by ICP-MS and QCM as a function of % LiOH ALD cycles.

D.J. Comstock, and J. W. Elam, *J. Phys. Chem. C* **117** (2013) 1677.



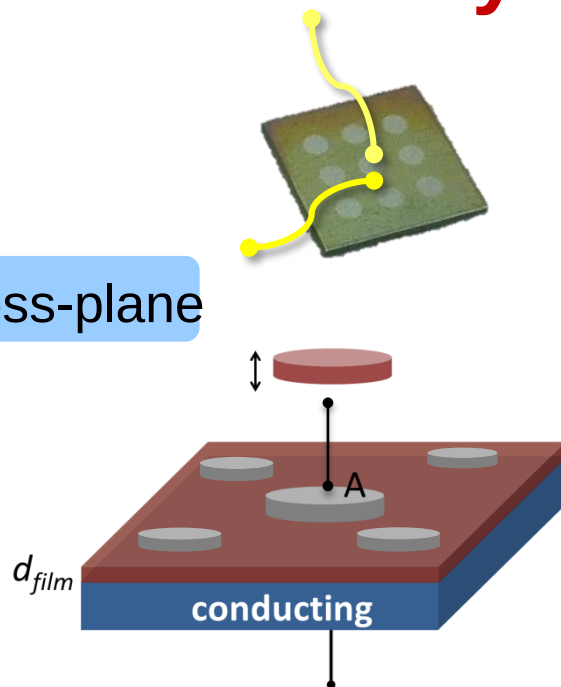
Lithium
trimethylsilanolate





Conductivity measurements

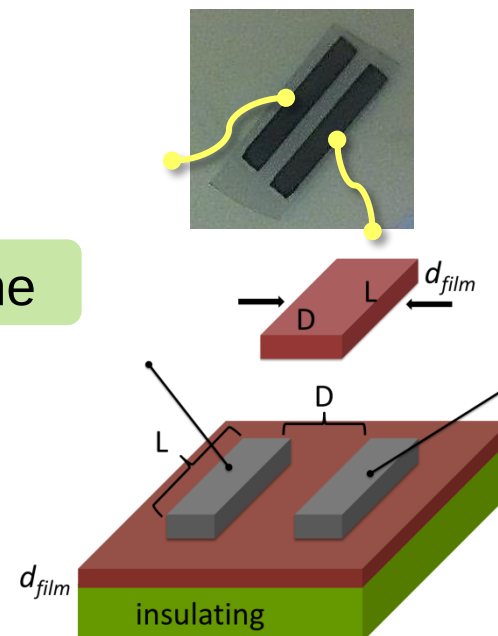
Cross-plane



$$\sigma_{cross} = \frac{L}{R \times A} = \frac{d_{film}}{R \times A_{electrode}}$$

- More practical interests
- Challenges: short-circuiting
- Difficult to carry out

In-plane



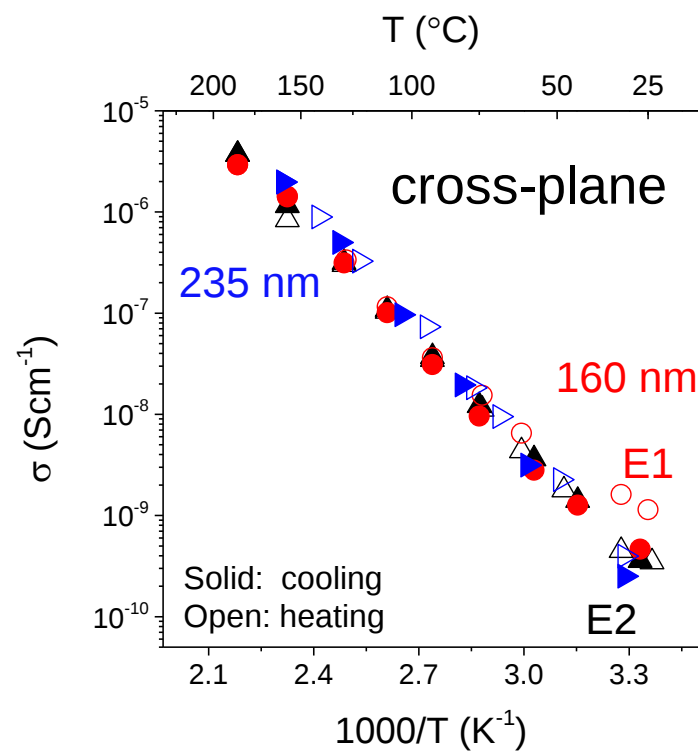
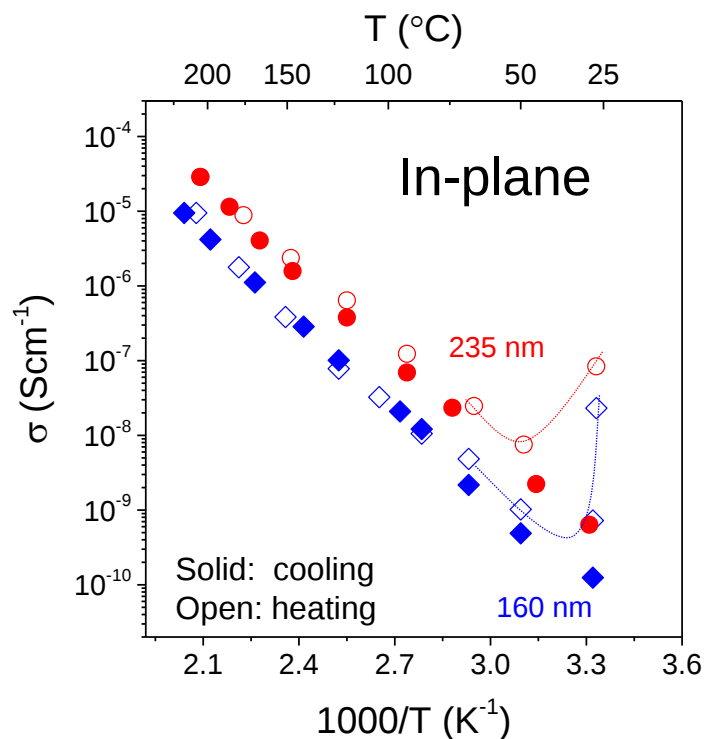
$$\sigma_{in} = \frac{L}{R \times A} = \frac{D_{electrode}}{R \times (d_{film} \times L)}$$

- Circumvent the short-circuitings
- Significant resistance
- More sensitive to parasitics





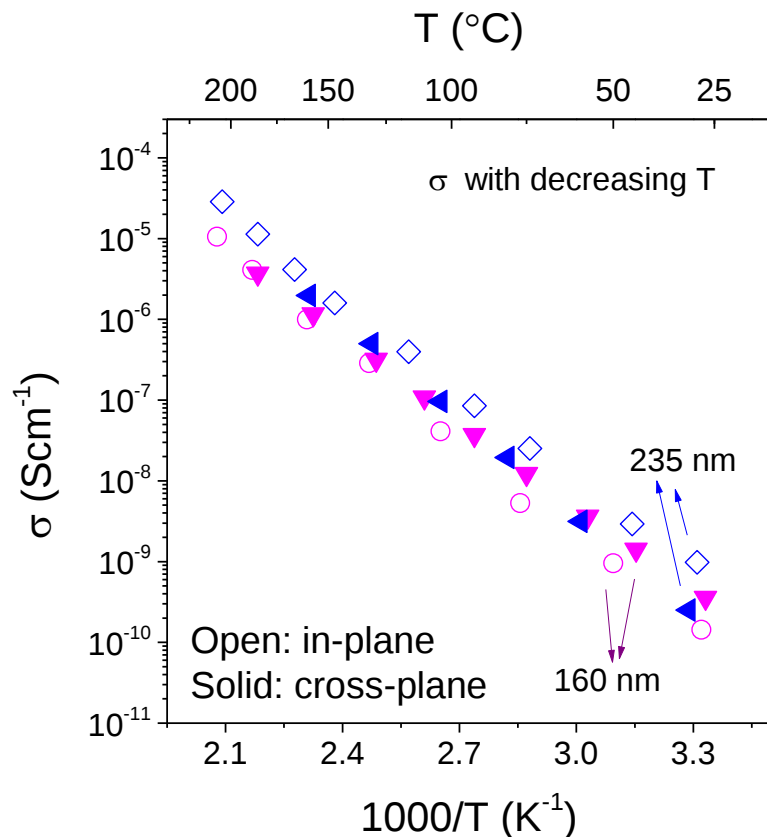
Conductivity of LiAlO₂ films



- Thermally activated ionic characteristics → Arrhenius relation: $\sigma = \frac{\sigma_0}{T} \exp\left(-\frac{E_a}{kT}\right)$
- Larger thickness-dependence for in-plane method: *surface, interface*
- σ @ room temperature: 10⁻¹⁰ ~ 10⁻⁹ Scm⁻¹



Conductivity of LiAlO_2 films



Materials	σ_{RT} (S cm ⁻¹)	E_a (eV)	Ref.
Single-crystalline γ -LiAlO ₂	$\sim 1 \times 10^{-17}$ *	1.14(1)	1
Polycrystalline γ -LiAlO ₂	2×10^{-14} *	0.81 (extrinsic) 1.3 (intrinsic)	2
ALD LiAlO ₂ film on quartz substrate	5.6×10^{-8} *	0.56	3
Quenched glass 0.6Li ₂ O–0.4Al ₂ O ₃ 0.7Li ₂ O–0.3Al ₂ O ₃	3×10^{-11} * 5×10^{-8} *	0.88 0.57	4
ALD LiAlO ₂ films, sapphire and Ti substrates	$1 \sim 5 \times 10^{-10}$	0.7–0.8	This work

- Room temperature conductivity was rarely reported
- Disordered amorphous/glassy $\text{Li}_x\text{AlO}_y \rightarrow$ higher conductivity
- Improved conductivity can be expected with increasing Li content

1. *The Journal of Physical Chemistry C* **2012**, 116, 14243
3. *J. Chem. Mater.* **2014**, 26, 3128-3134.

2. *J. Am. Ceram. Soc.* **1984**, 67, 418-41
4. *J. Appl. Phys.* **1980**, 51, 3756-3761.

Conclusion

Cathode:

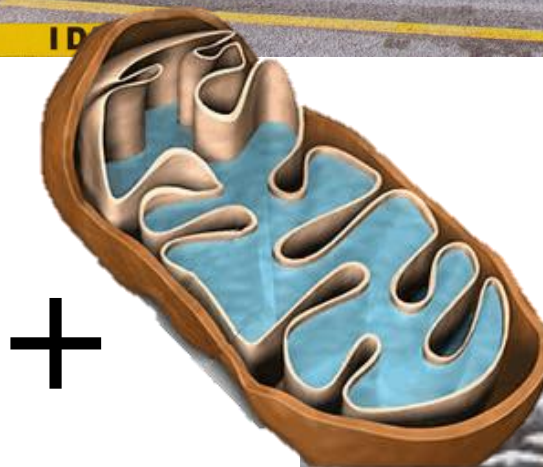
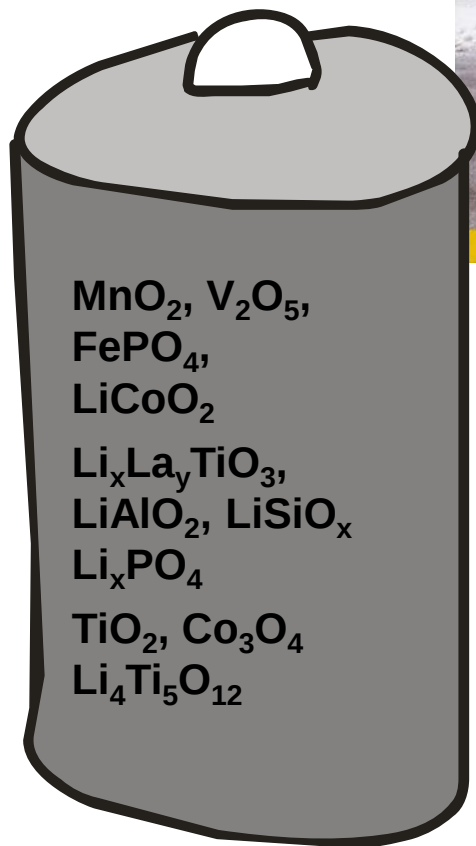
MnO_2 , V_2O_5 ,
 FePO_4 ,
 LiCoO_2

Electrolyte:

$\text{Li}_x\text{La}_y\text{TiO}_3$,
 LiAlO_2 , LiSiO_x
 Li_xPO_4

Anode:

TiO_2 , Co_3O_4
 $\text{Li}_4\text{Ti}_5\text{O}_{12}$



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