X-ray Diffraction Studies on the Lithiation of LiAl Electrodes for Li Ion Batteries

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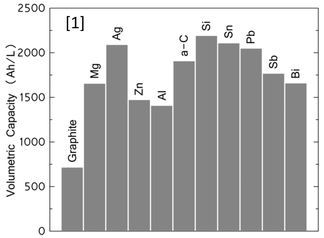
Aluminium as Anode Material

Why Aluminium?

- Higher volumetric capacity than graphite
- Large resources → low material costs

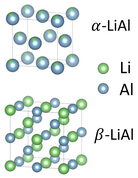


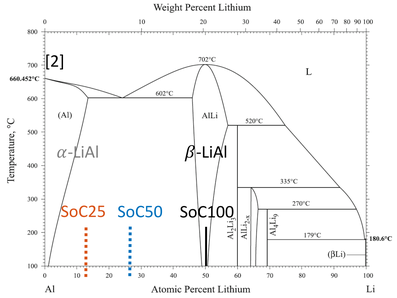
Samples

- Coin cells with Al sheets (16µm thickness) with Li counter electrode and electrolyte (1.2M LiPF6, EC:EMC 3:7 + 10wt% FEC)
- Disassembled and washed with DMC in glovebox

Phase Diagram

- α-LiAl: Fm-3m
  - Solid solution of Li and Al
  - Same crystal structure as pristine Al
- β-LiAl: Fd-3m
  - Nucleation of β-particles once solubility limit of Li in Al reached

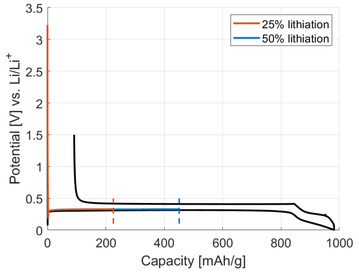




Experimental Procedure

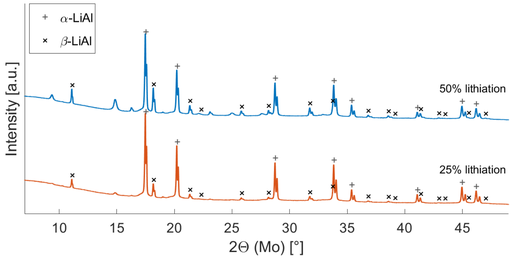
Lithiation

- Performed in 40°C temperature chamber
- Cells loaded with C/40 to SoC25 and SoC50 (β-LiAl = fully lithiated phase = SoC100)



X-Ray Measurements

- LiAl samples in sealed Kapton foil
- Measured with Mo radiation in transmission mode for higher penetration



Results from X-Ray Diffraction Measurements

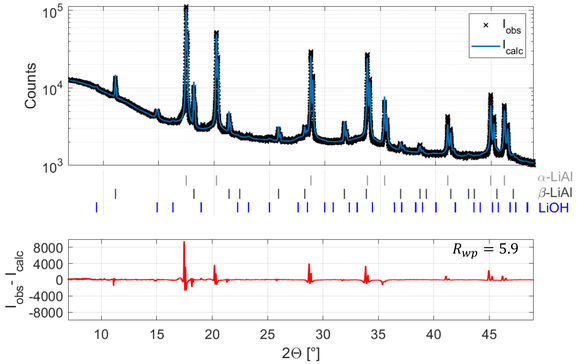
Rietveld Refinement

Identified Phases:

- ✓ α-LiAl
- ✓ β-LiAl
- ✓ LiOH

expected from phase diagram

unexpected

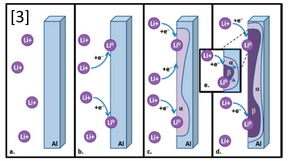


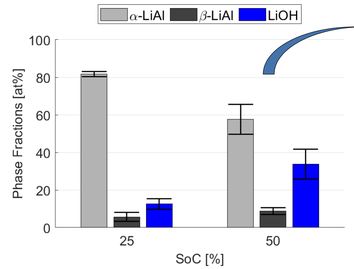
Phase Fractions

- Higher α-LiAl for 25% lithiation
- Higher β-LiAl and LiOH for 50% lithiation

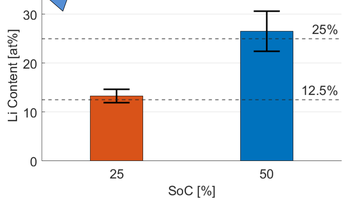
Formation of LiOH

- Samples exposed to ambient air
- Reaction of Li with moisture





Assumption [4]:
5.5% Lithiation of α-LiAl phase



Outlook

Operando XRD Measurements

- Study phase transformations during lithiation
- Correlation with potential curve → Determination of phase trainstion onset
- Reduction of ambient air contamination → Inhibition of LiOH formation

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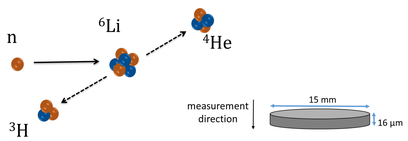
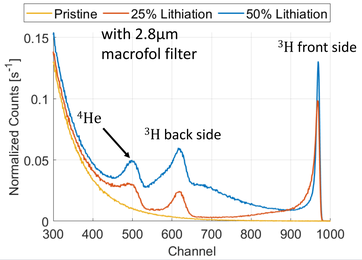
NDP Measurements at CANAM in Řež

- Determination of Li concentration
- Estimation of depth of β-LiAl particles





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