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Decoupling electrolyte and electrode reactions using in-operando electrochemical x-ray powder diffraction

Oles Sendetskyi[1,2,*]

Mark Salomons[2],
Steve Launspach[2],
Michael Fleischauer[1,2]

[1]*Department of Physics, University of Alberta, Edmonton, AB T6G 2E1, Canada*
[2]*National Research Council - Nanotechnology Research Centre, Edmonton, AB T6G 2M9, Canada*

* *sendetsk@ualberta.ca*

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Li-ion batteries

- Dominant energy storage technology

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* pixabay, unsplash

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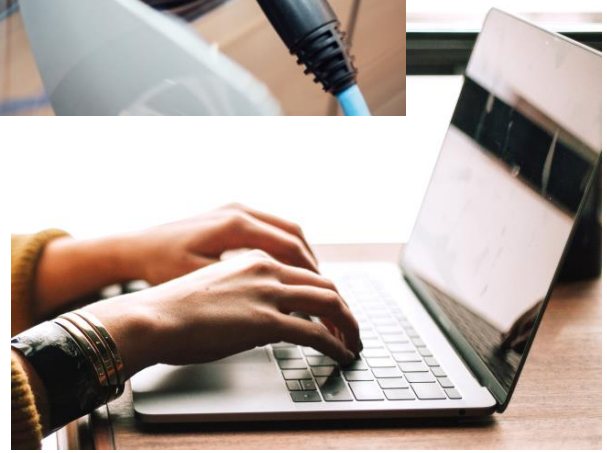

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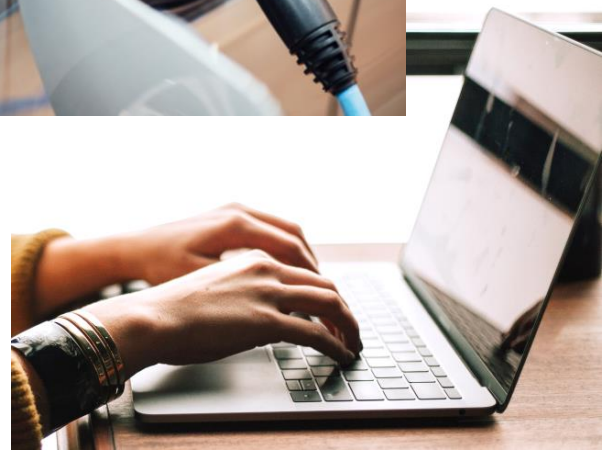
Li-ion batteries

- Dominant energy storage technology
- Increase capacity and reduce cost using novel electrodes, like alkali-metal alloys



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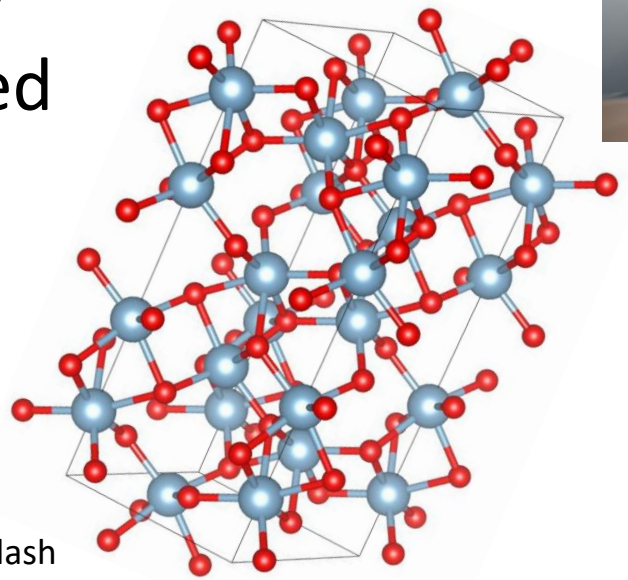
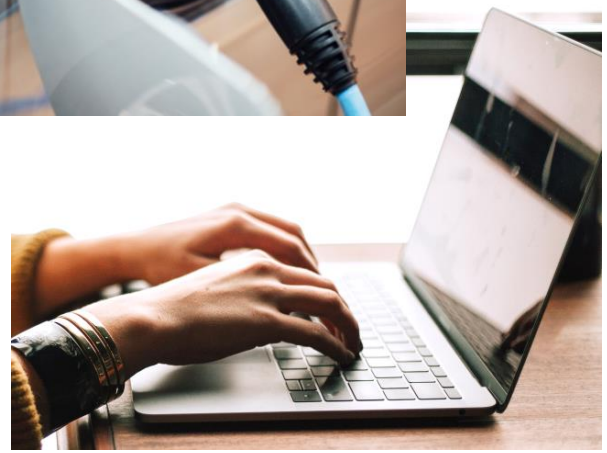
- Dominant energy storage technology
- Increase capacity and reduce cost using novel electrodes, like alkali-metal alloys
- Quantitative analysis of battery electrode composition is needed





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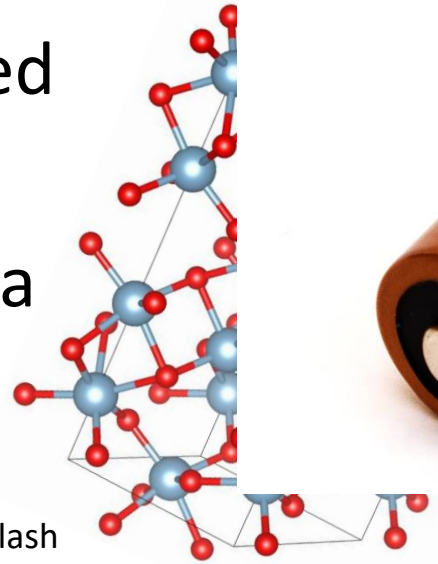
Li-ion batteries

- Dominant energy storage technology
- Increase capacity and reduce cost using novel electrodes, like alkali-metal alloys
- Quantitative analysis of battery electrode composition is needed
- *In-situ* and *in-operando* X-ray diffraction on Li-ion cells using a versatile Conflat cell with a Beryllium window



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Li-ion cells with Al electrodes

Benefits of Al electrodes:

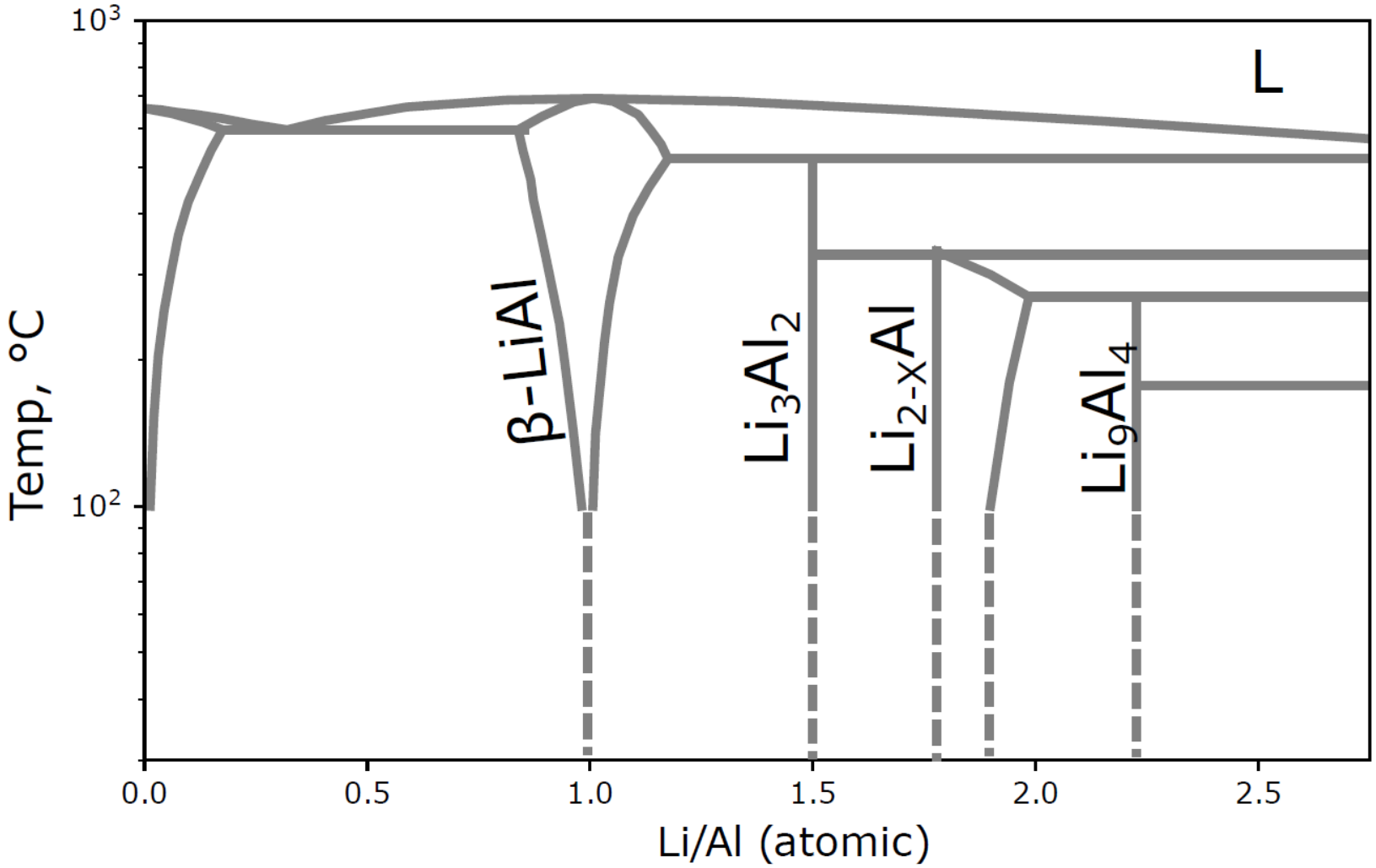
- Offer high capacity ≈ 2000 mAh/g, 6x graphite
- Low cost
- Rich Al-Li phase diagram for in-operando testing
- Testing in wide temperature range

Problems of Al electrodes:

- Capacity fading
- Slow diffusion and nucleation barriers

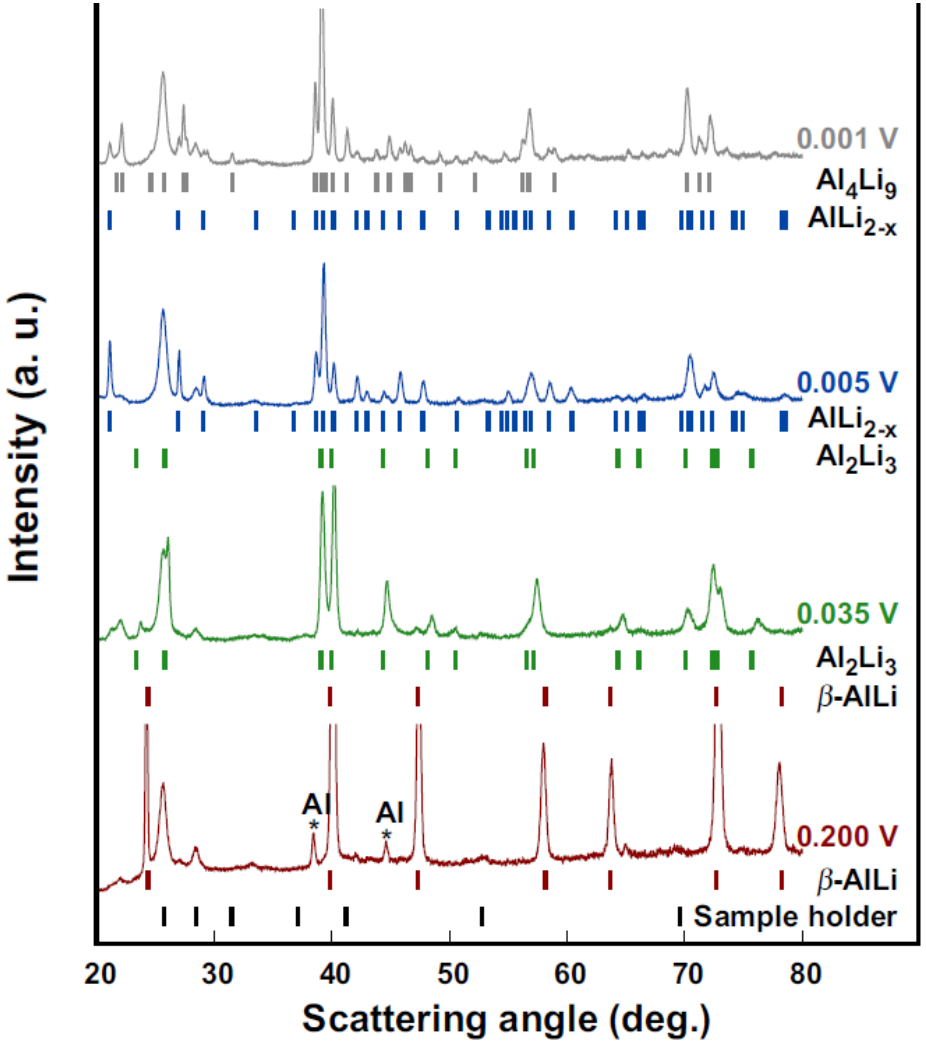
H. Li et al, Nature Communications, 11, 1584 (2020)
M. Z. Ghavidel et al, Journal of The Electrochemical Society, 166 (16) A4034-A4040 (2019)

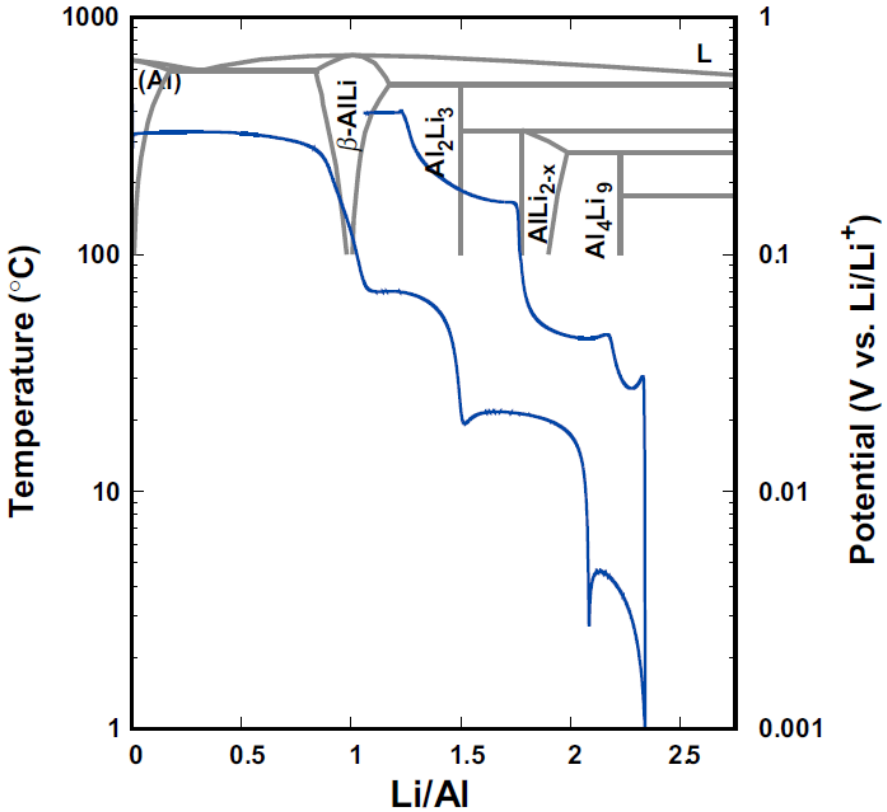
Li-Al has many equilibrium phases (β-LiAl, Li_3Al_2, $Li_{2-x}Al$, Li_9Al_4)



after ASM Binary Phase Diagrams

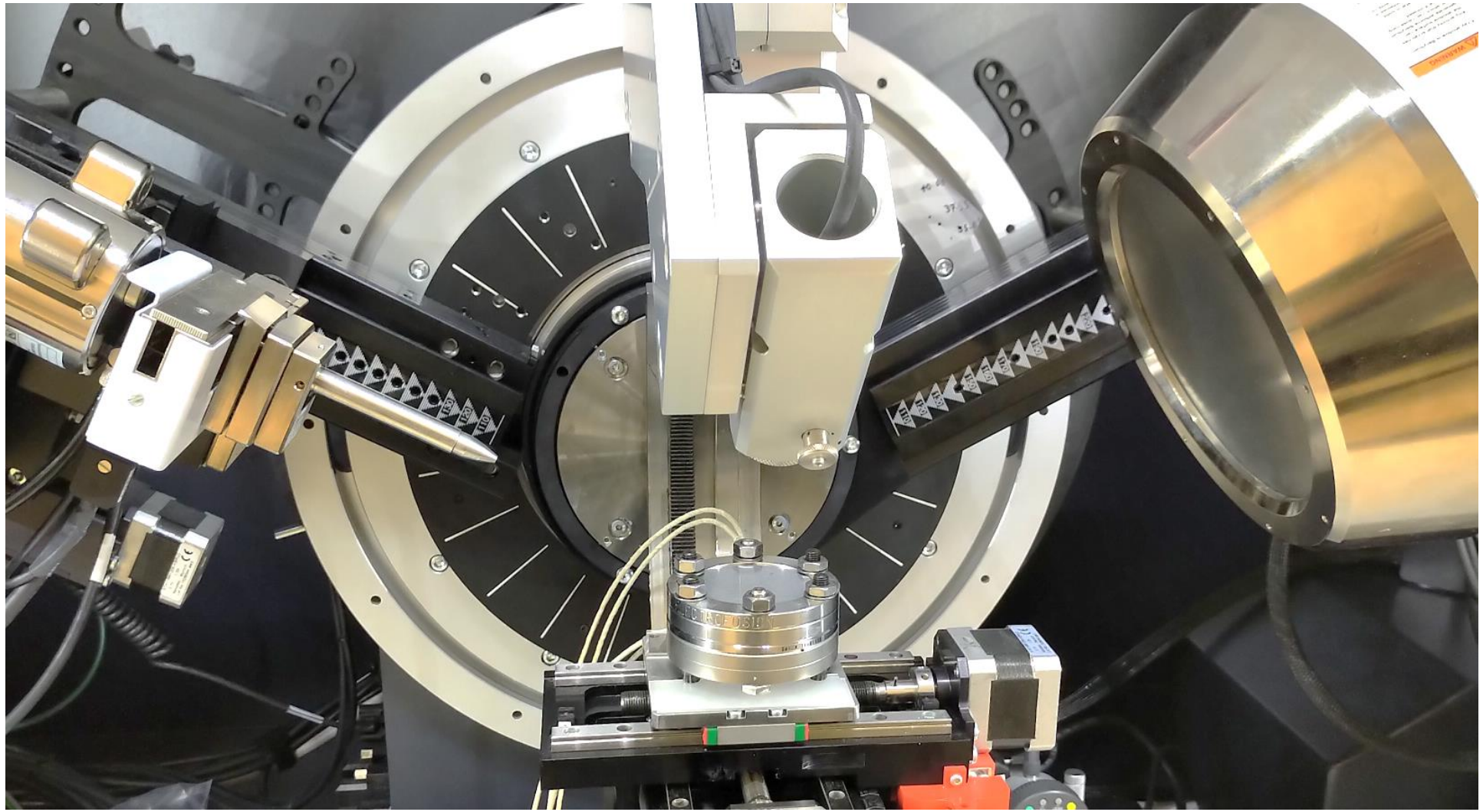
Ex-situ Al-Li measurements



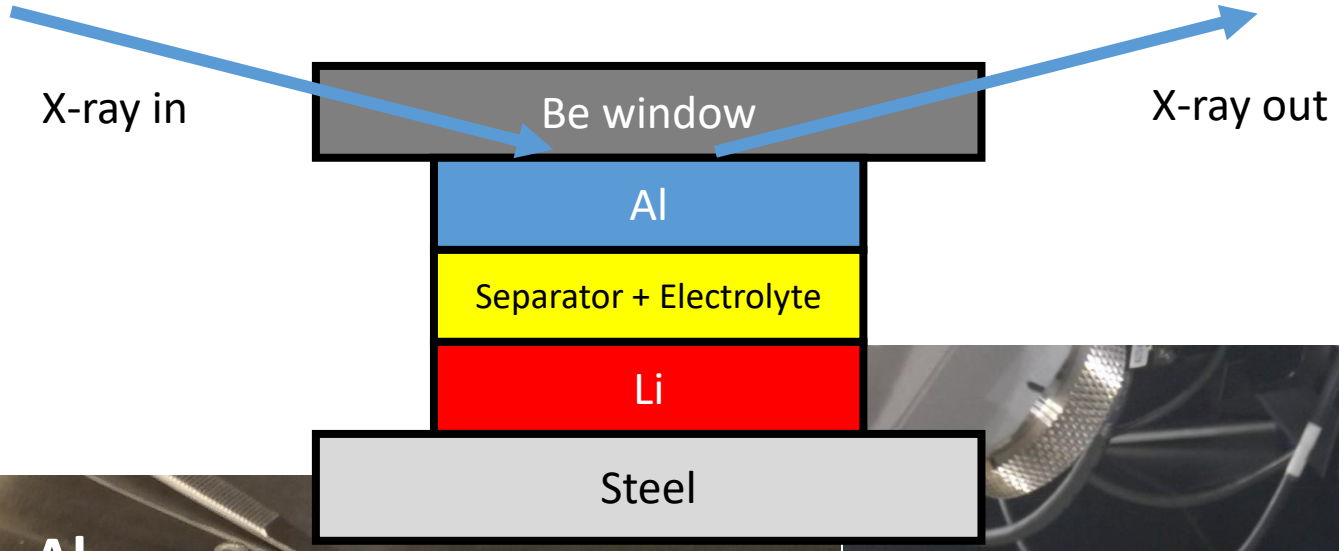


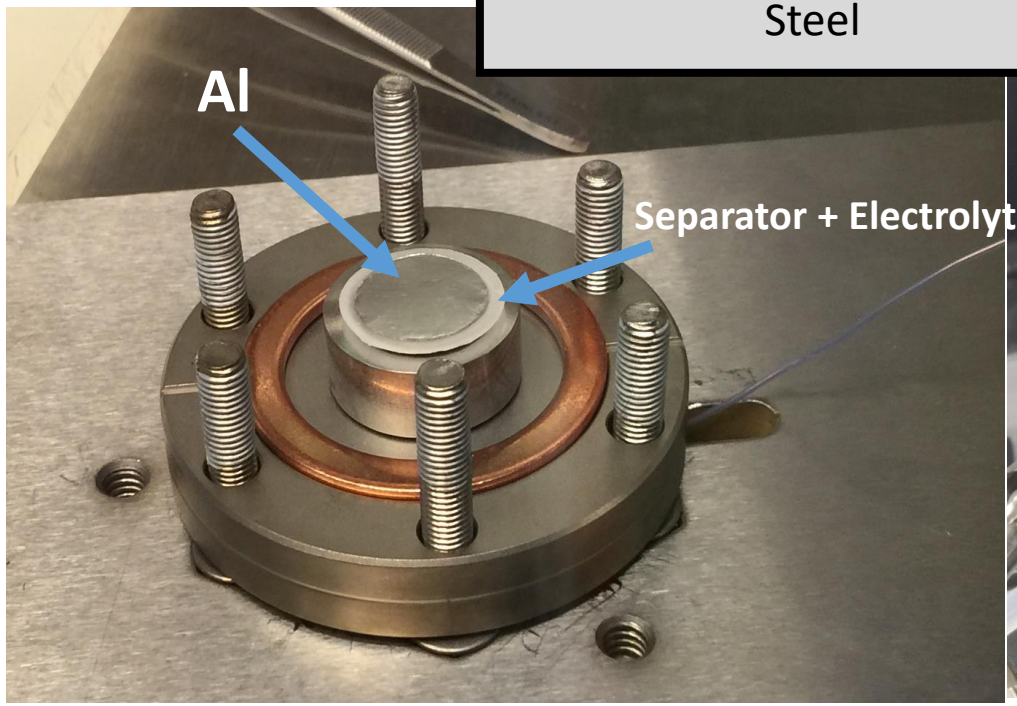
M. Z. Ghavidel et al, Journal of The Electrochemical Society, 166 (16) A4034-A4040 (2019)

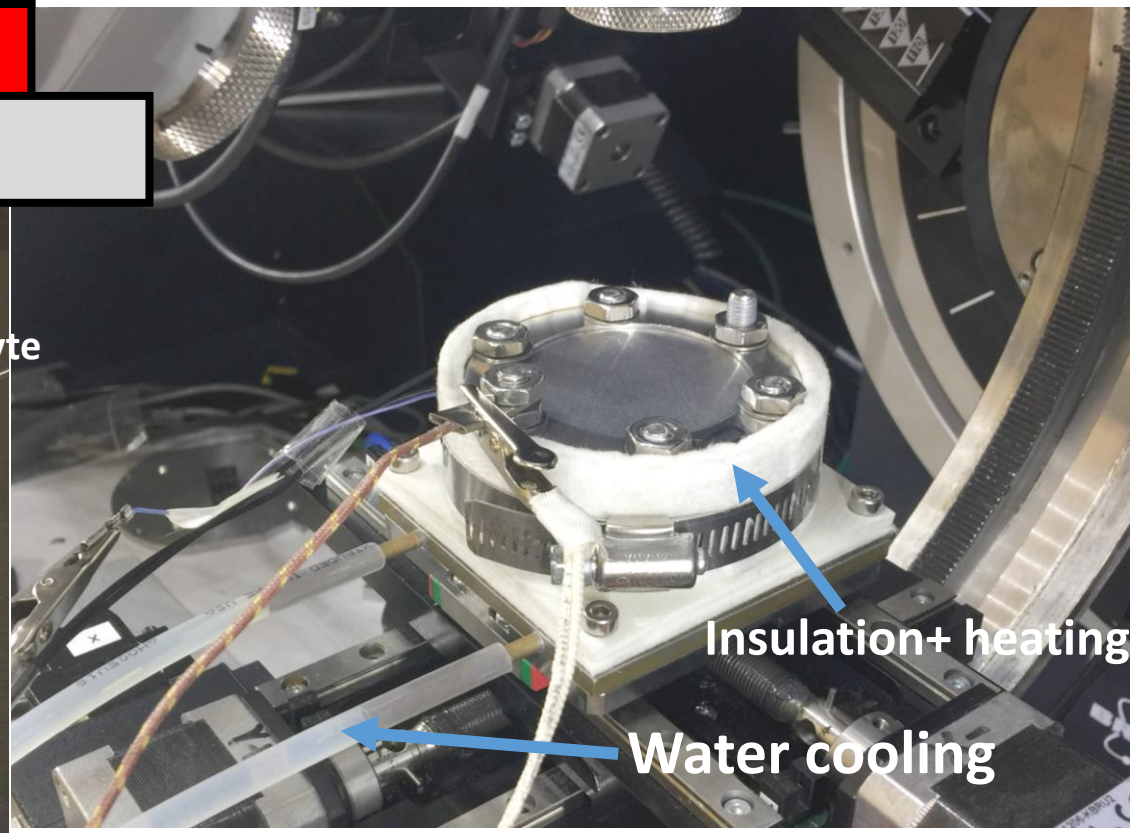
In-situ XRD setup, Bruker D8



In-situ XRD cell and high temperature setup

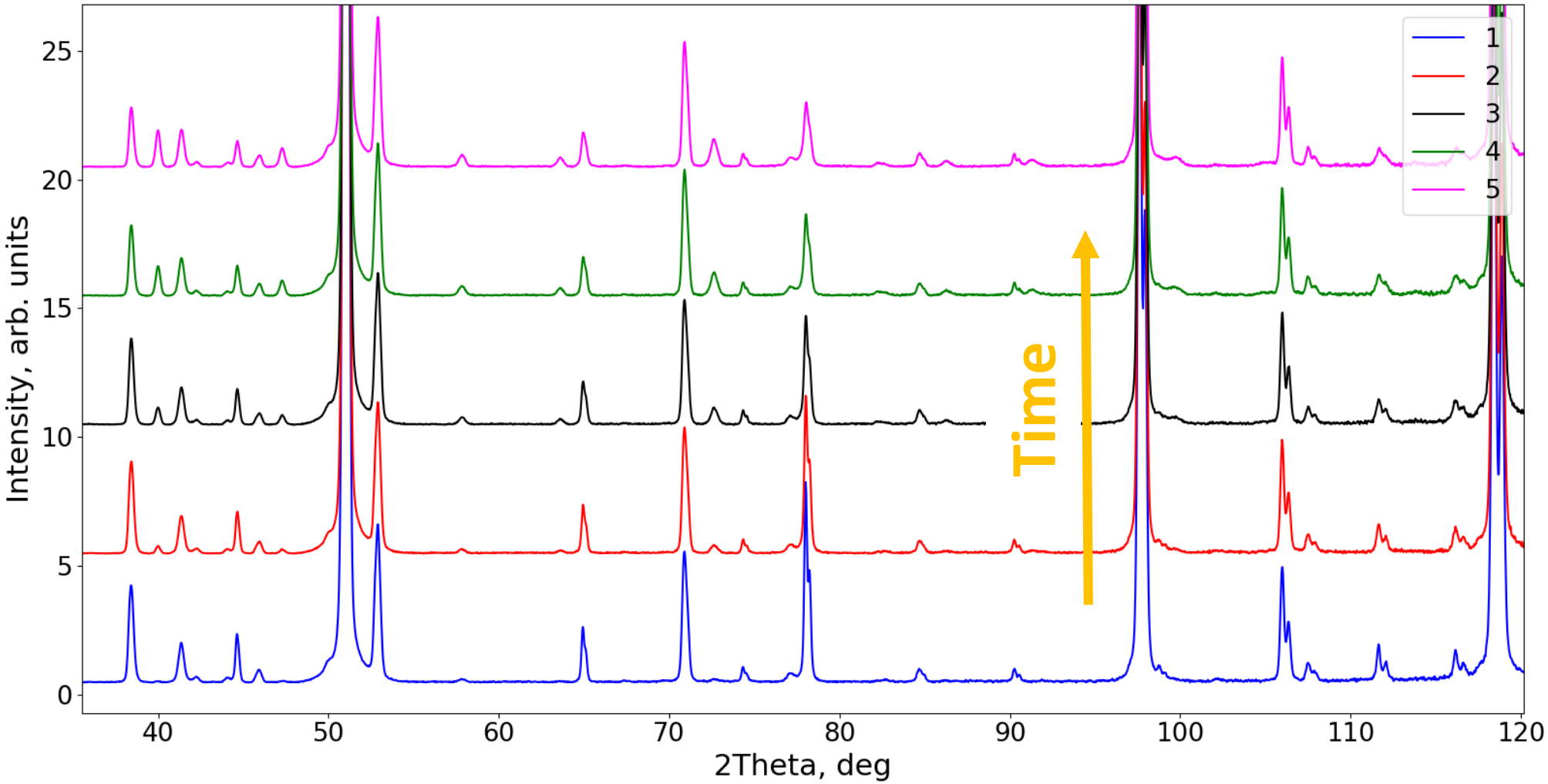






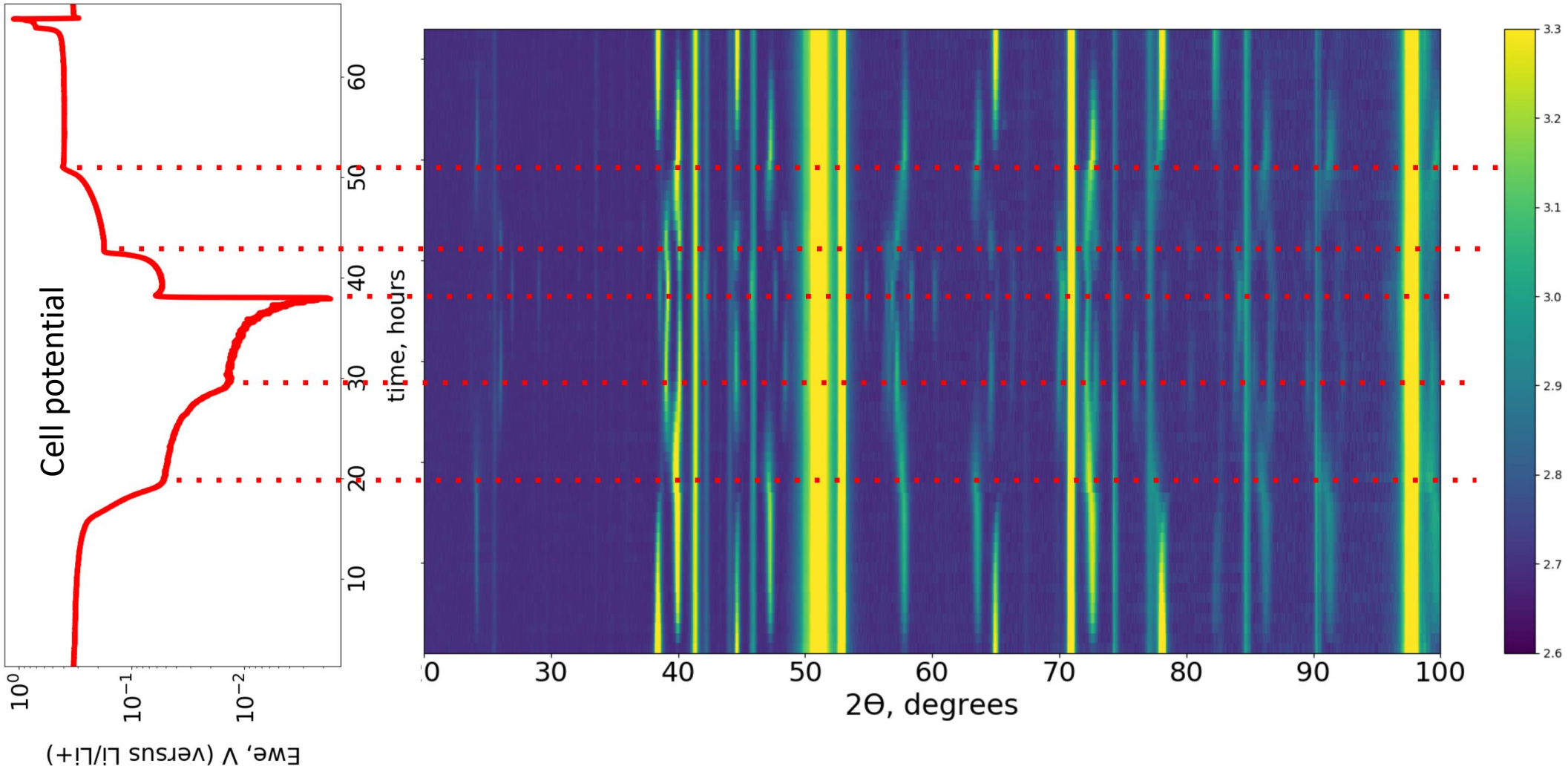
In-situ XRD measurements

XRD patterns are continuously measured one after another.



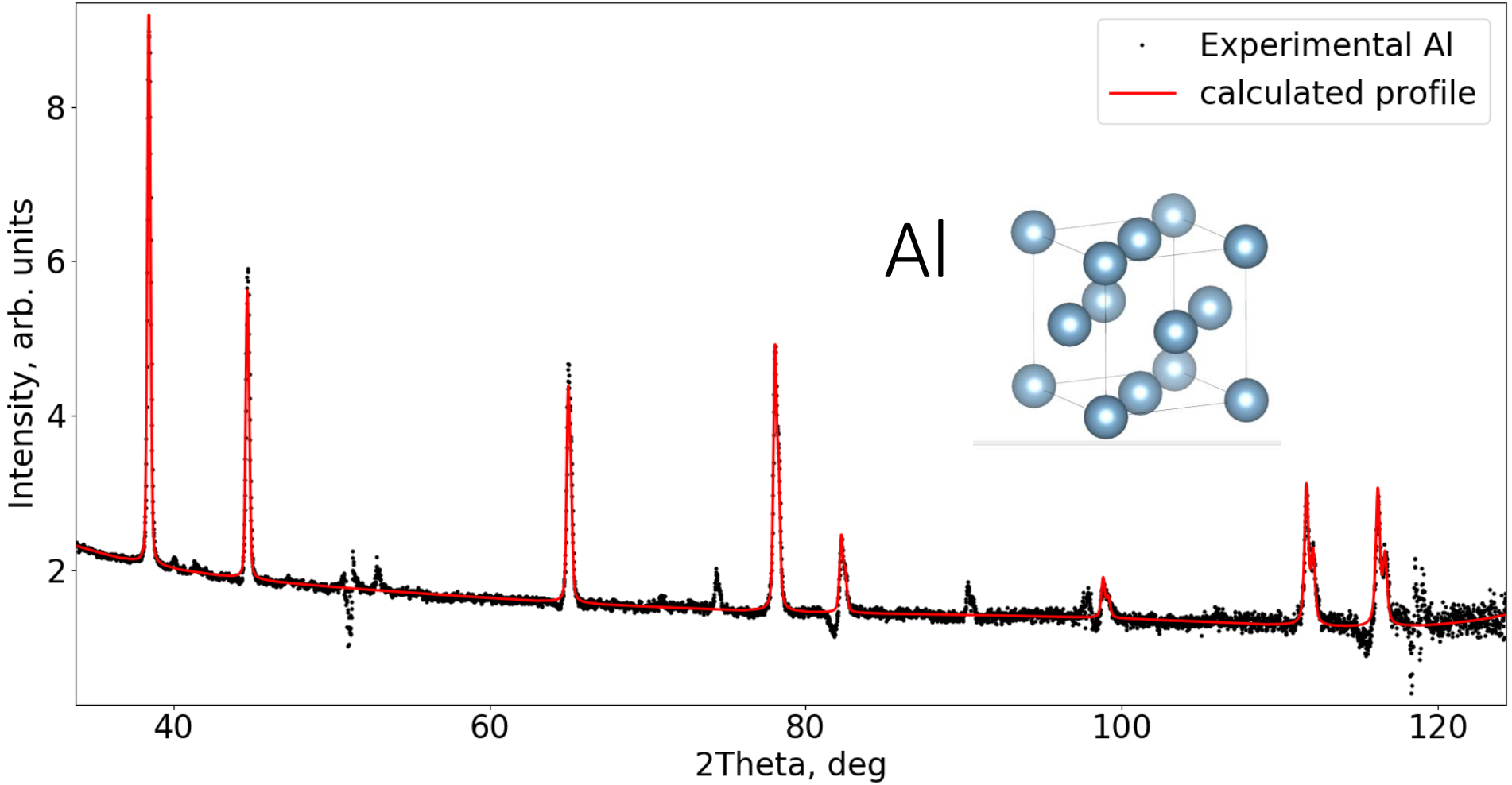
XRD pattern evolution

In order to get reliable composition of phases at each potential, structure refinement is needed.



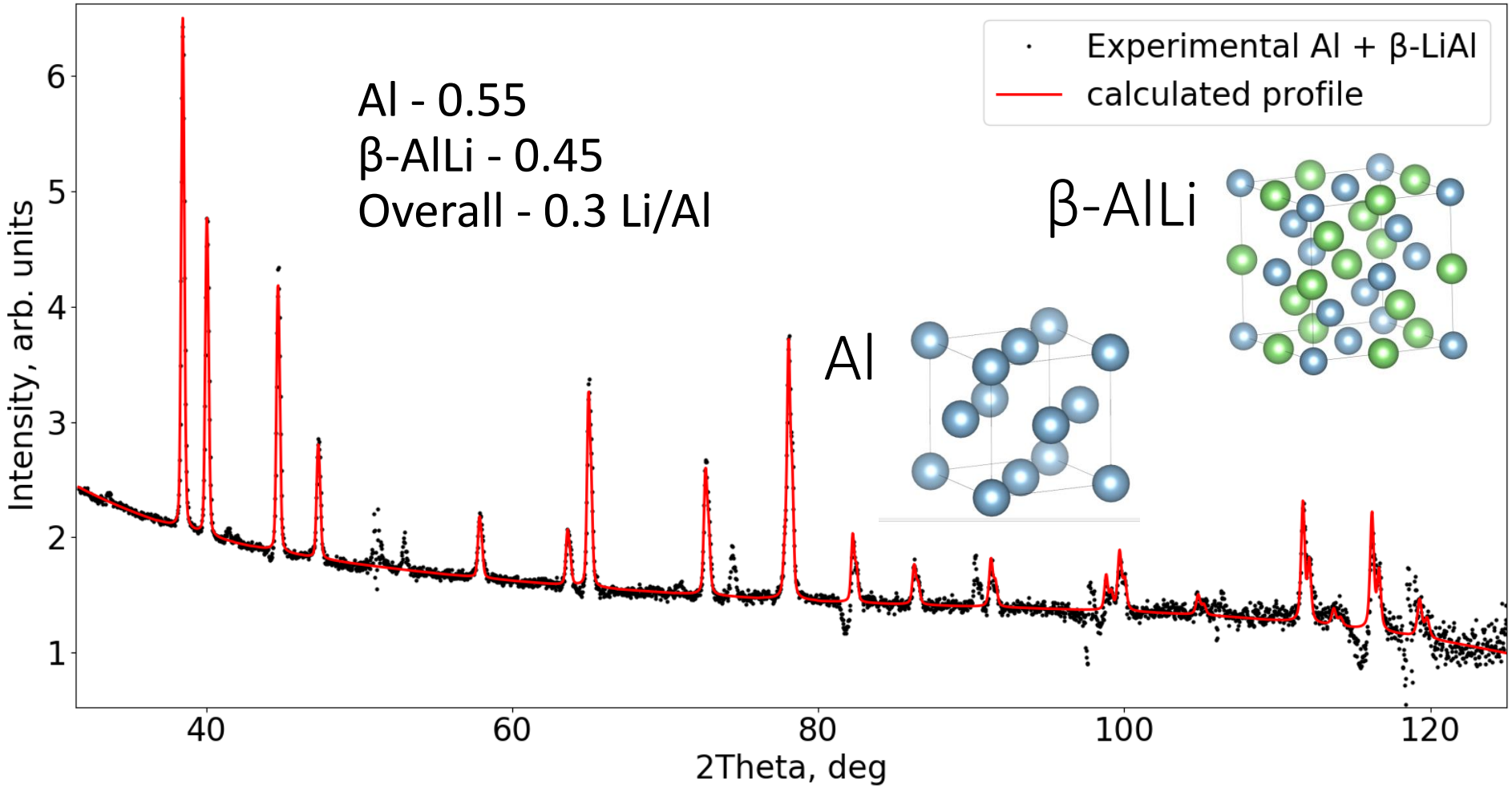
Single phase refinement - Al

Measured through the Be window, then Be signal is subtracted and a clean Al pattern is refined.



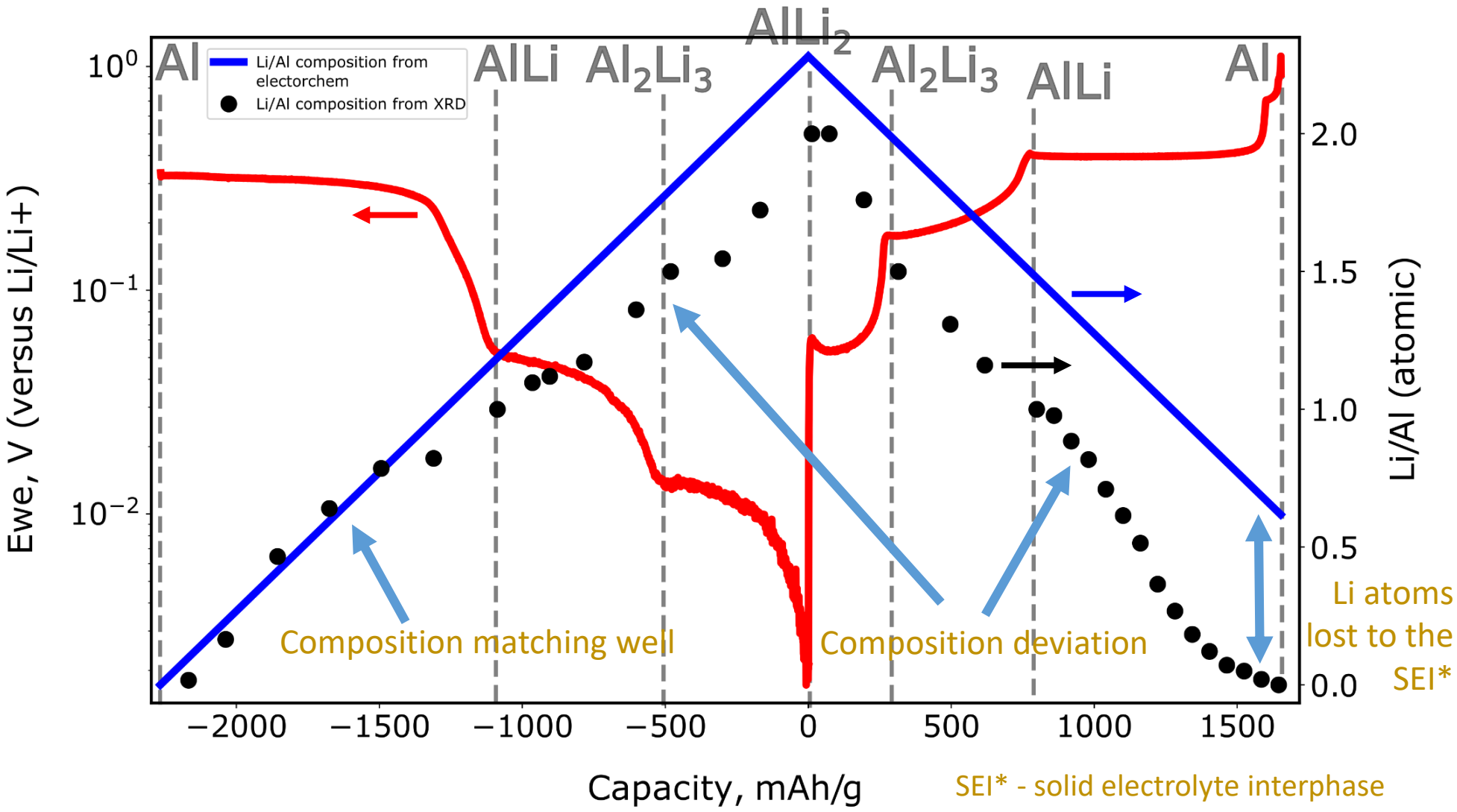
Multiphase refinement – Al / β-AlLi

Multiphase refinement of Al and β-AlLi mixture.



Electrode composition

Blue line – composition calculated from the cell current.
Black dots – composition obtained from structure refinements.



SEI* - solid electrolyte interphase

Acknowledgements

The authors would like to acknowledge funding from the National Research Council Canada and University of Alberta Nanotechnology Initiative.

Conclusions

- Lab based in-operando / in-situ

electrochemical XRD

- Conflat cell allows probing wide range of temperatures
- Pattern refinement allows to quantify the composition of the electrode
- Significant difference in the composition highlights electrolyte breakdown during the first cycle

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