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

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**L05-3177** October 4-9 2020, PRiME 2020, Hawaii

# Li-ion batteries

- Dominant energy storage technology

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# Li-ion batteries

- Dominant energy storage technology



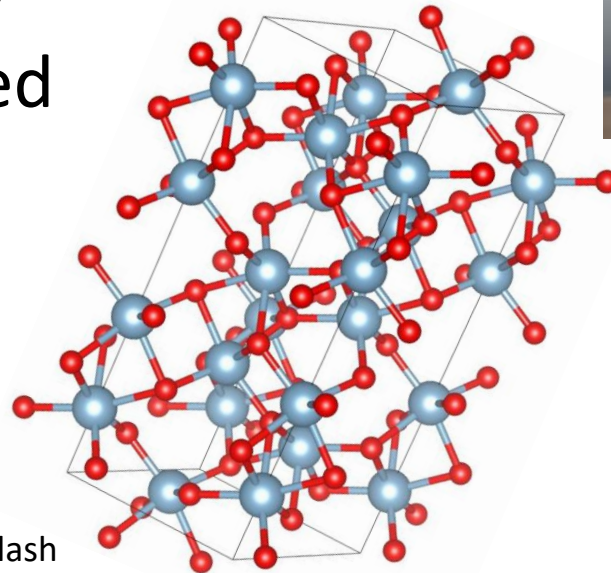
# Li-ion batteries

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



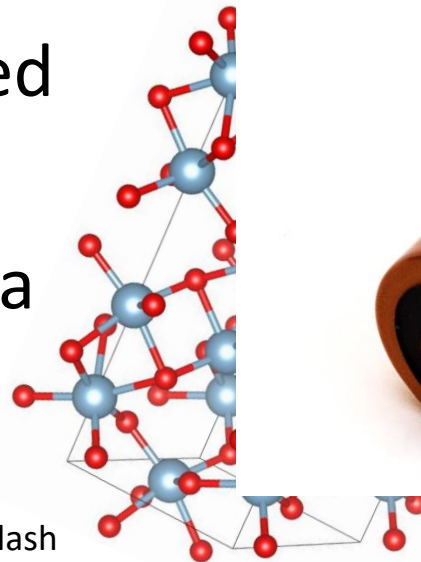
# Li-ion batteries

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



# 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



\* pixabay, unsplash



# Li-ion cells with Al electrodes

## Benefits of Al electrodes:

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

## Problems of Al electrodes:

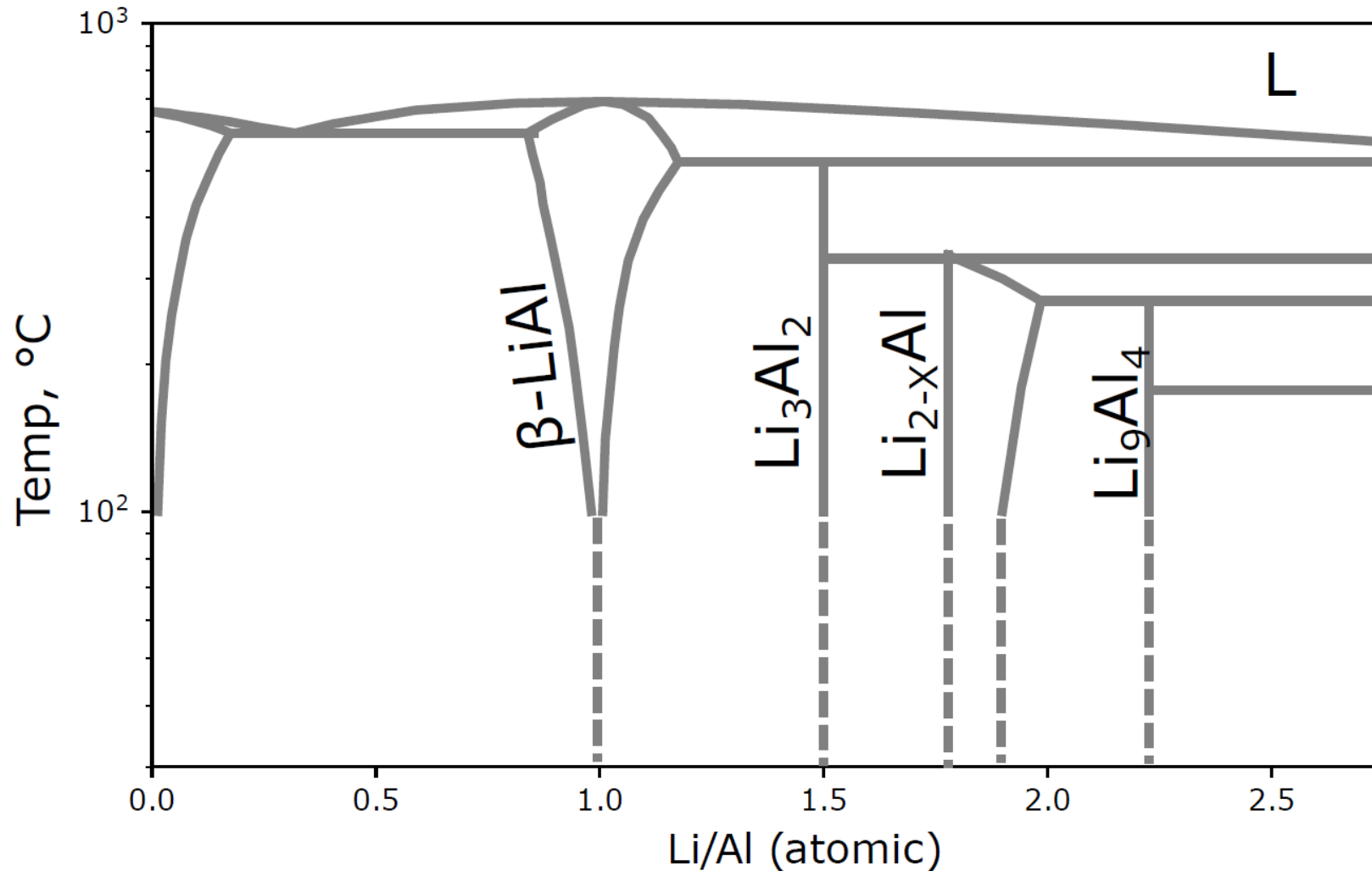
- Capacity fading
- Slow diffusion and nucleation barriers

H. Li et al, Nature Communications, 11, 1584 (2020)

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



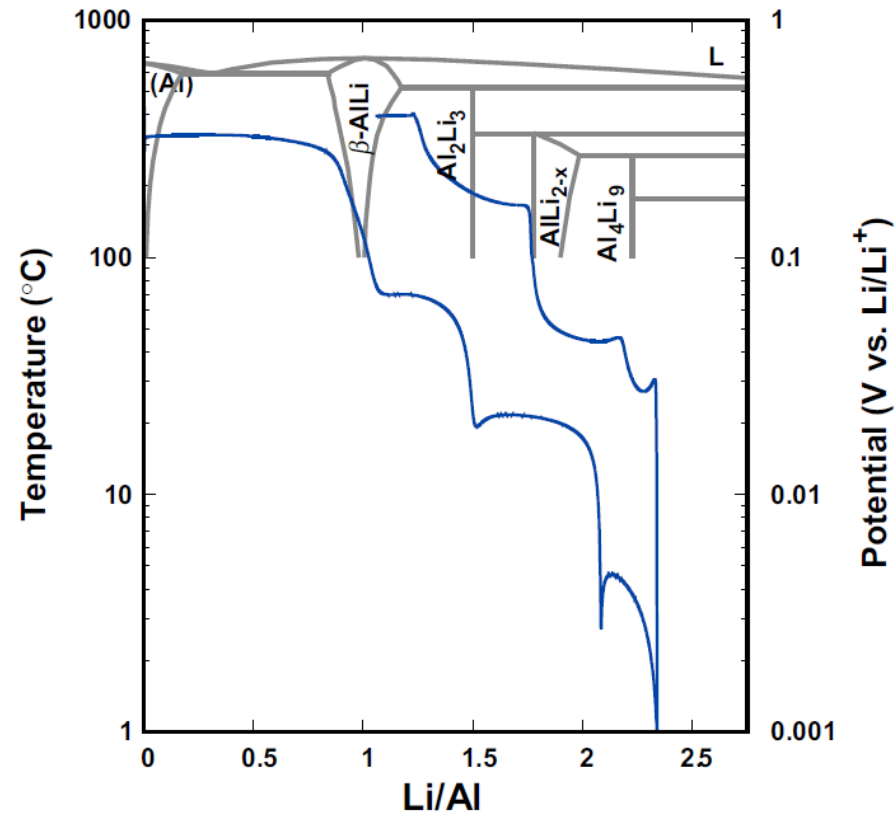
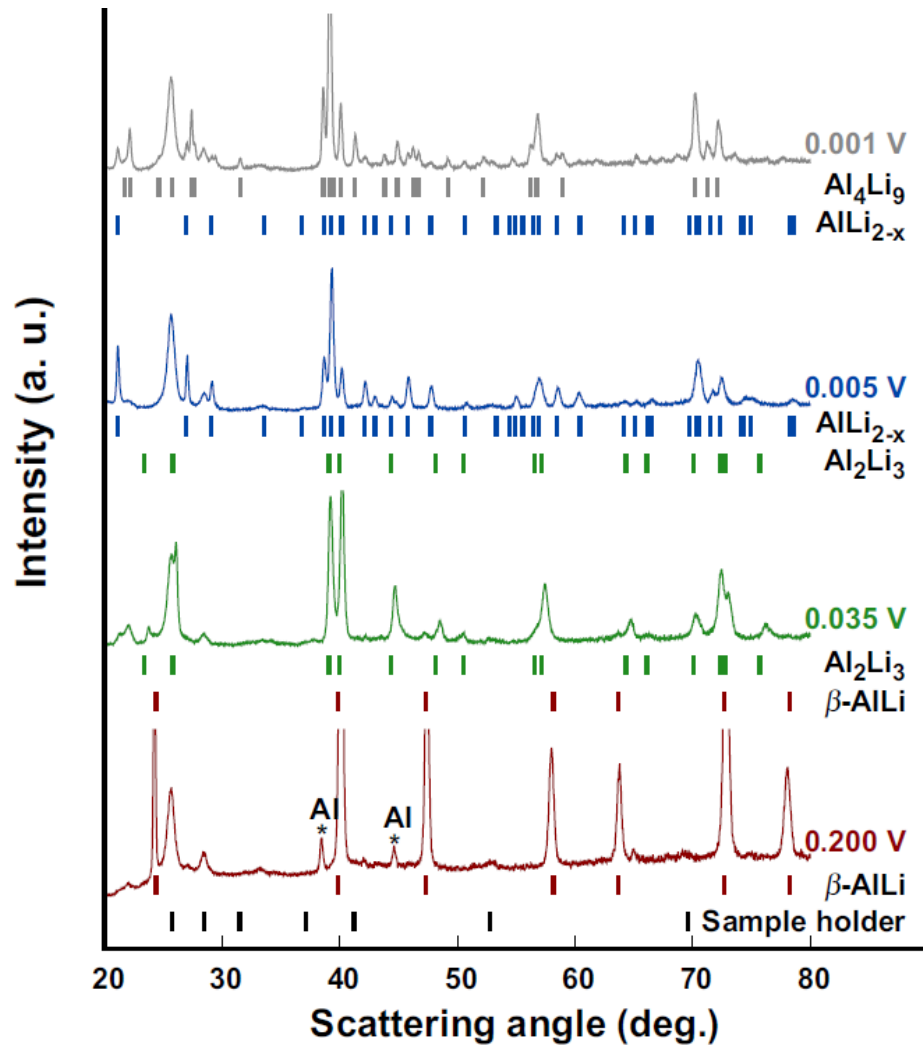
Li-Al has many equilibrium phases  
( $\beta$ -LiAl,  $\text{Li}_3\text{Al}_2$ ,  $\text{Li}_{2-x}\text{Al}$ ,  $\text{Li}_9\text{Al}_4$ )



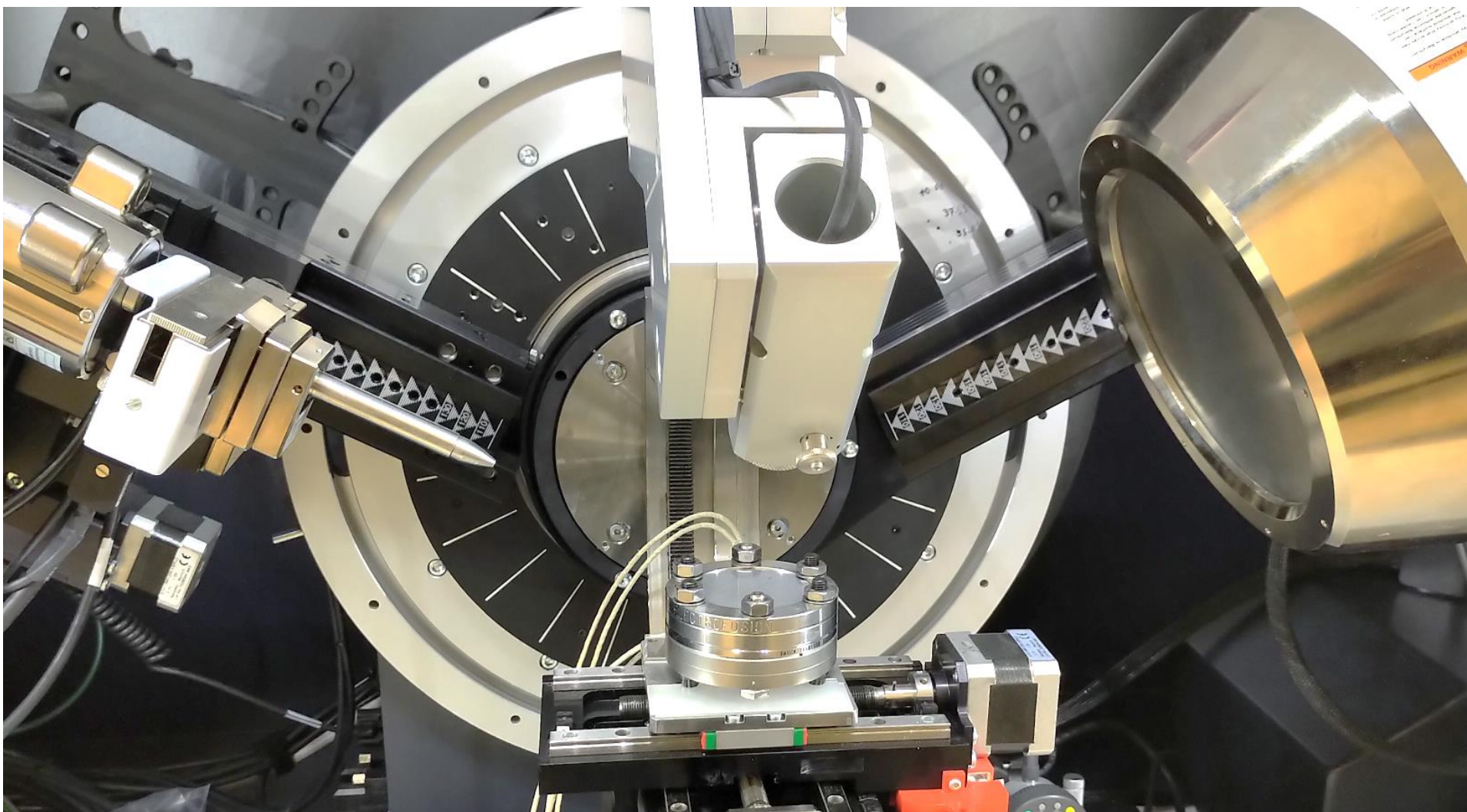
after ASM Binary Phase Diagrams



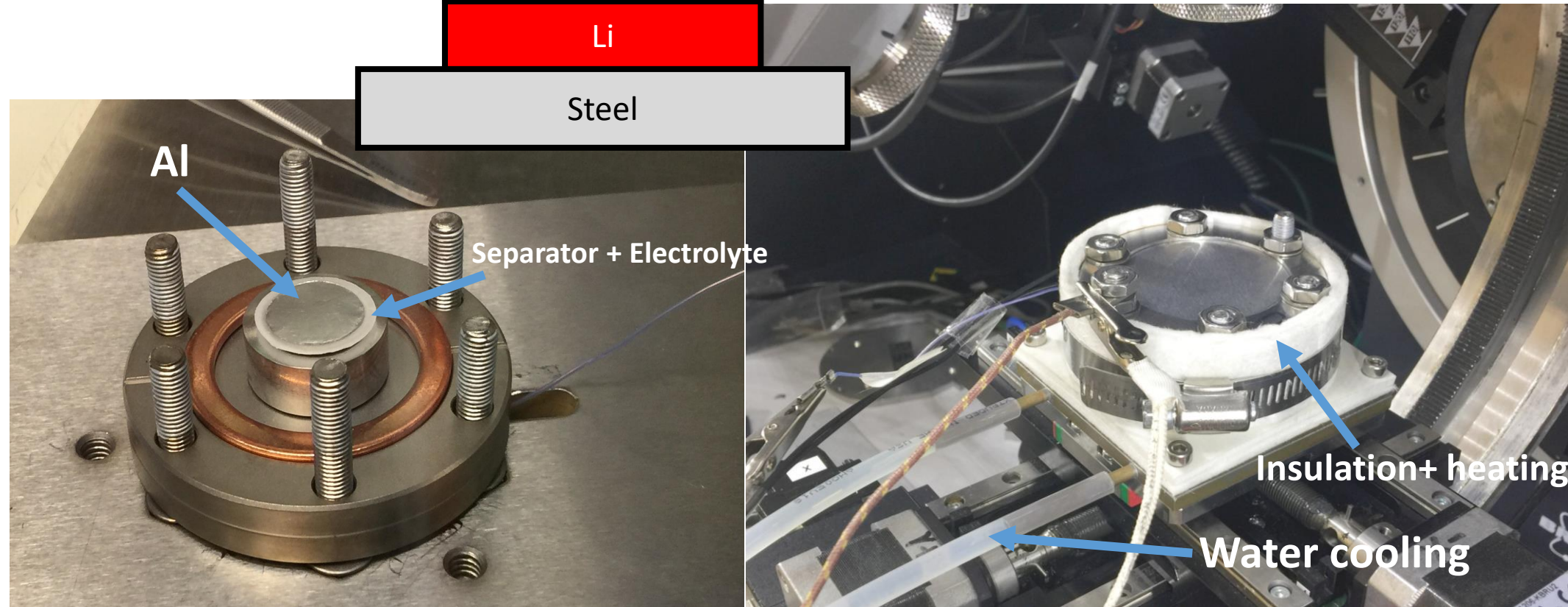
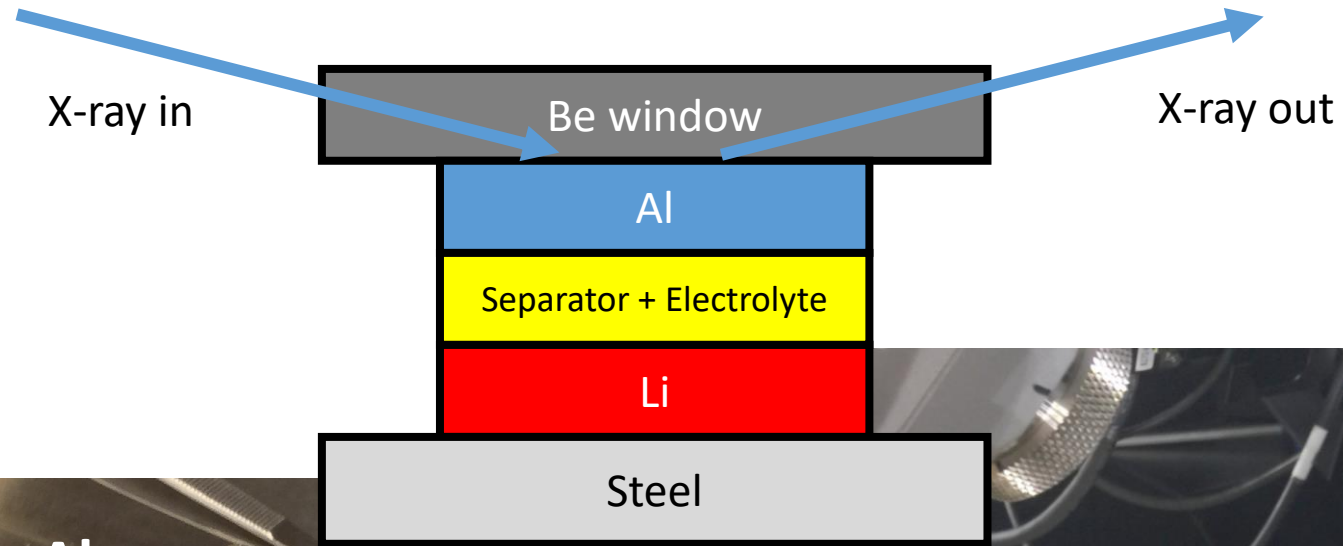
# Ex-situ Al-Li measurements



# In-situ XRD setup, Bruker D8

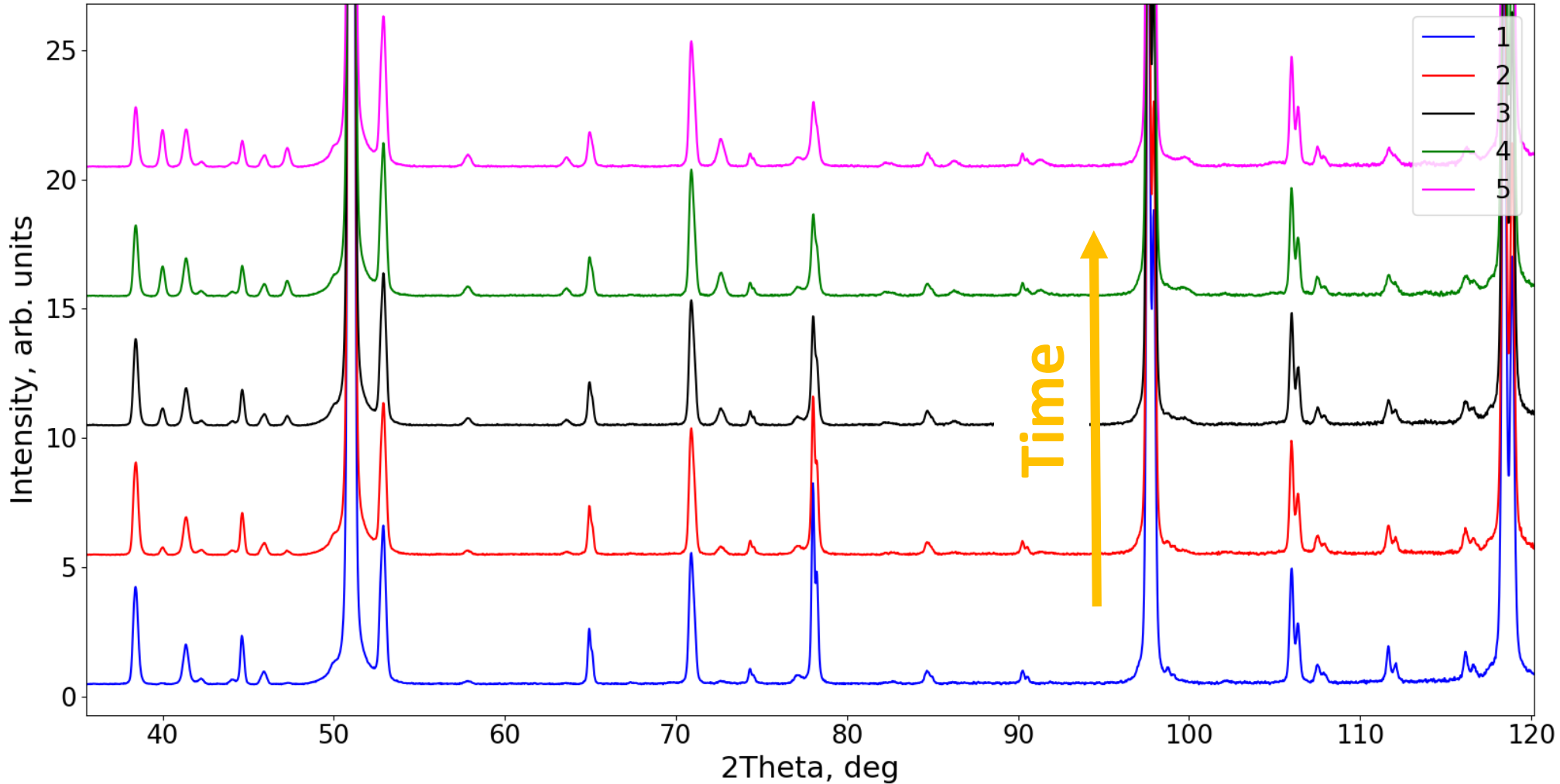


# In-situ XRD cell and high temperature setup



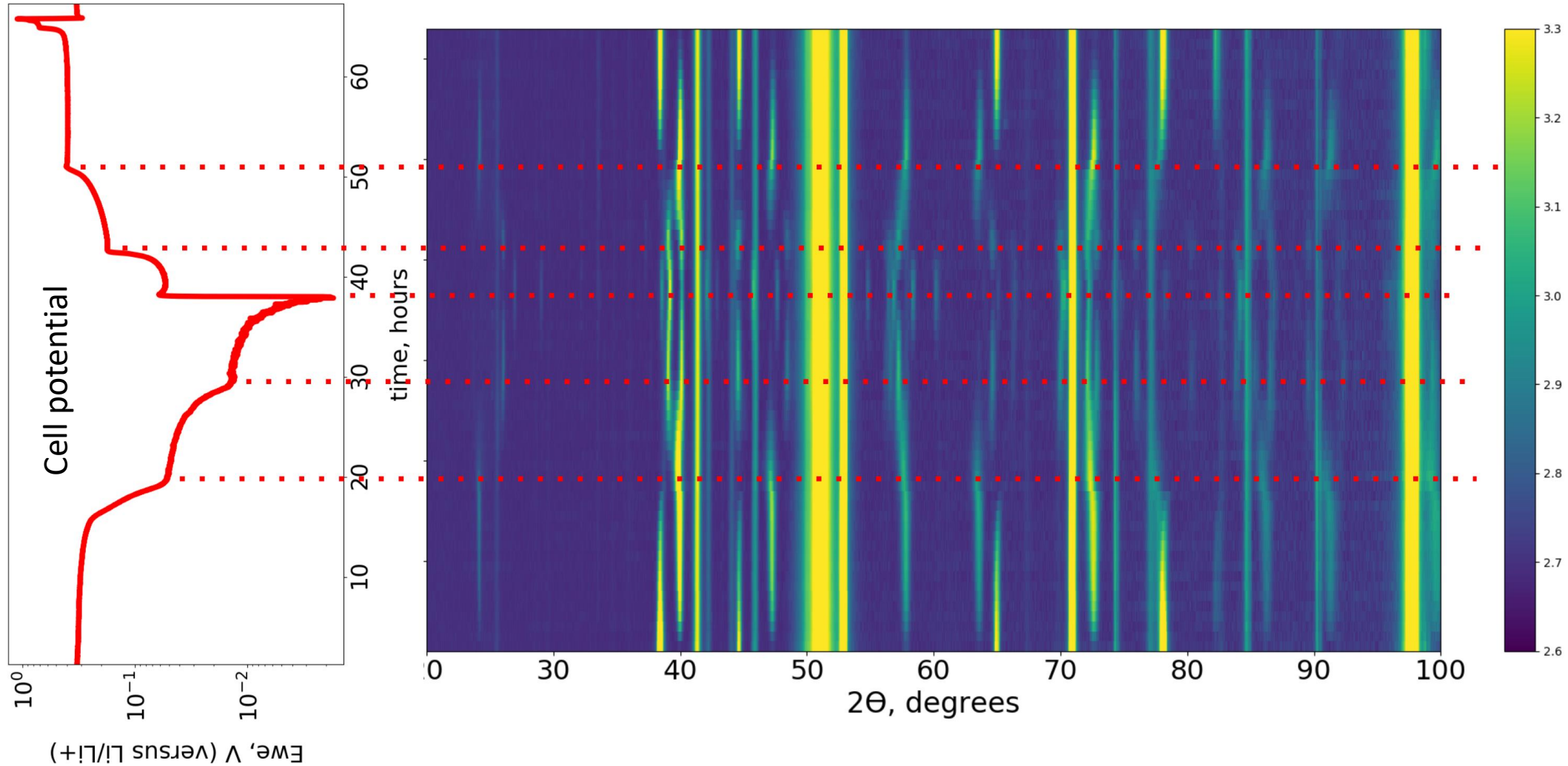
# In-situ XRD measurements

XRD patterns are continuously measured one after another.



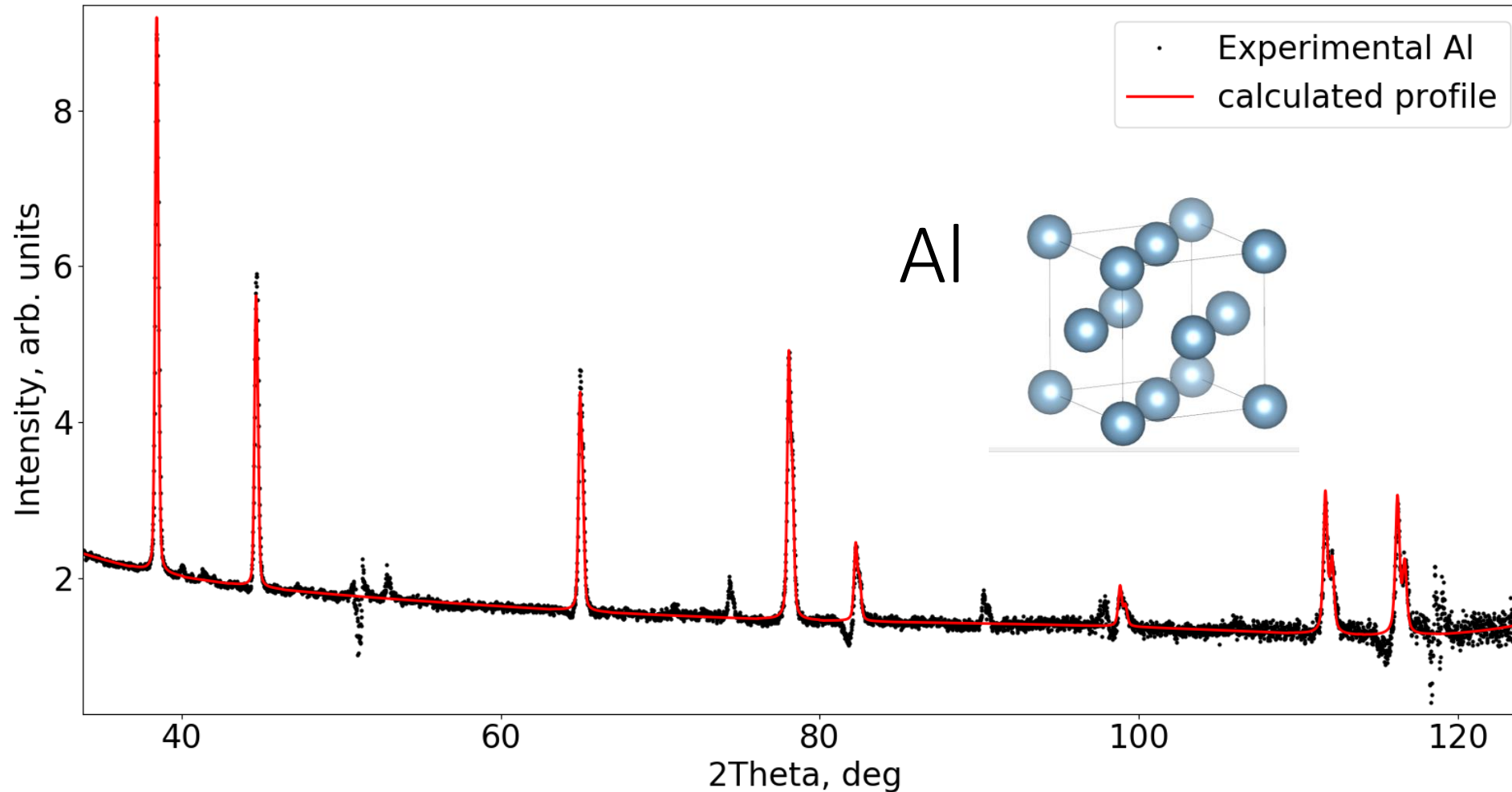
# XRD pattern evolution

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



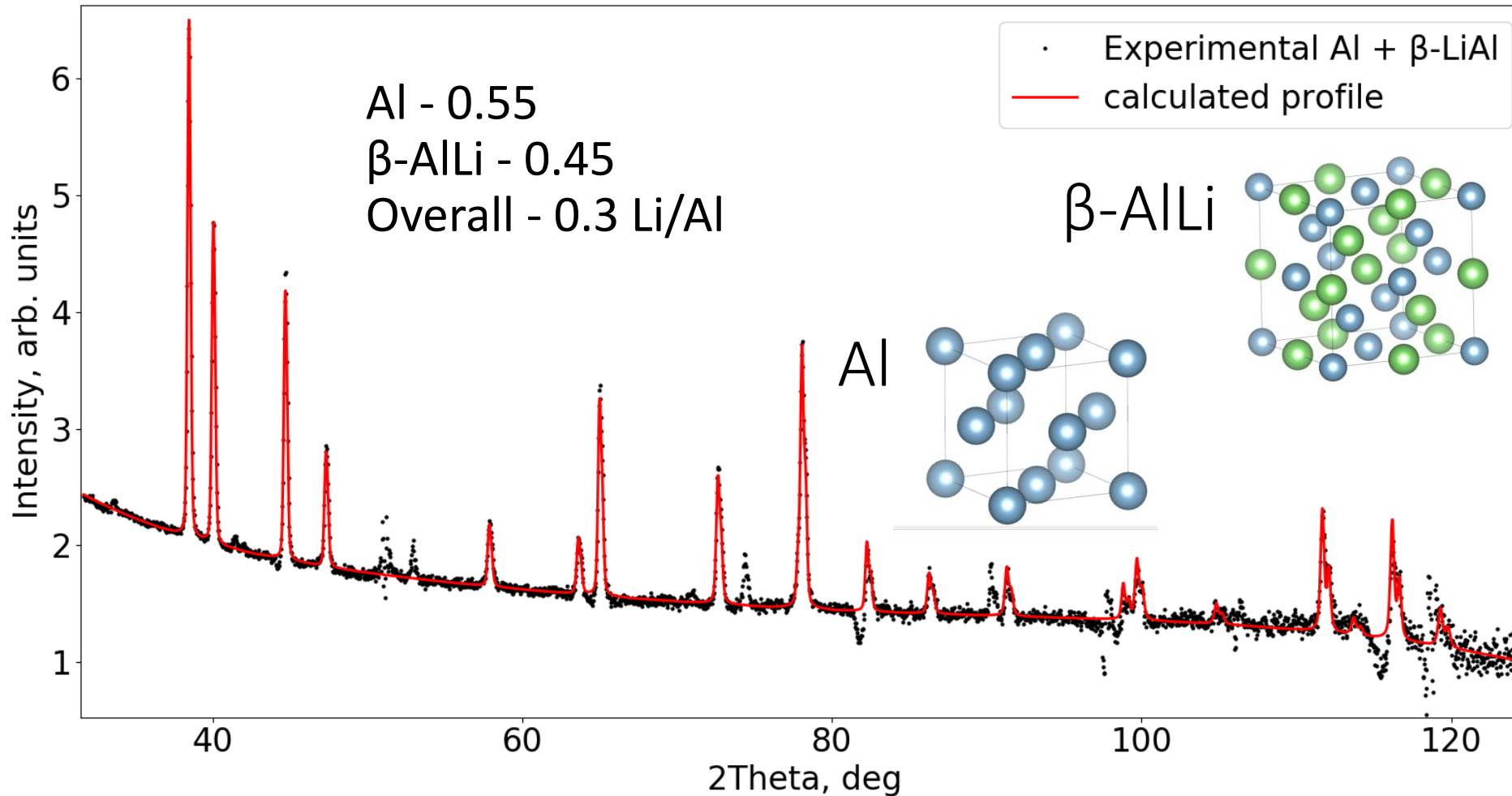
# Single phase refinement - Al

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



# Multiphase refinement – Al / $\beta$ -AlLi

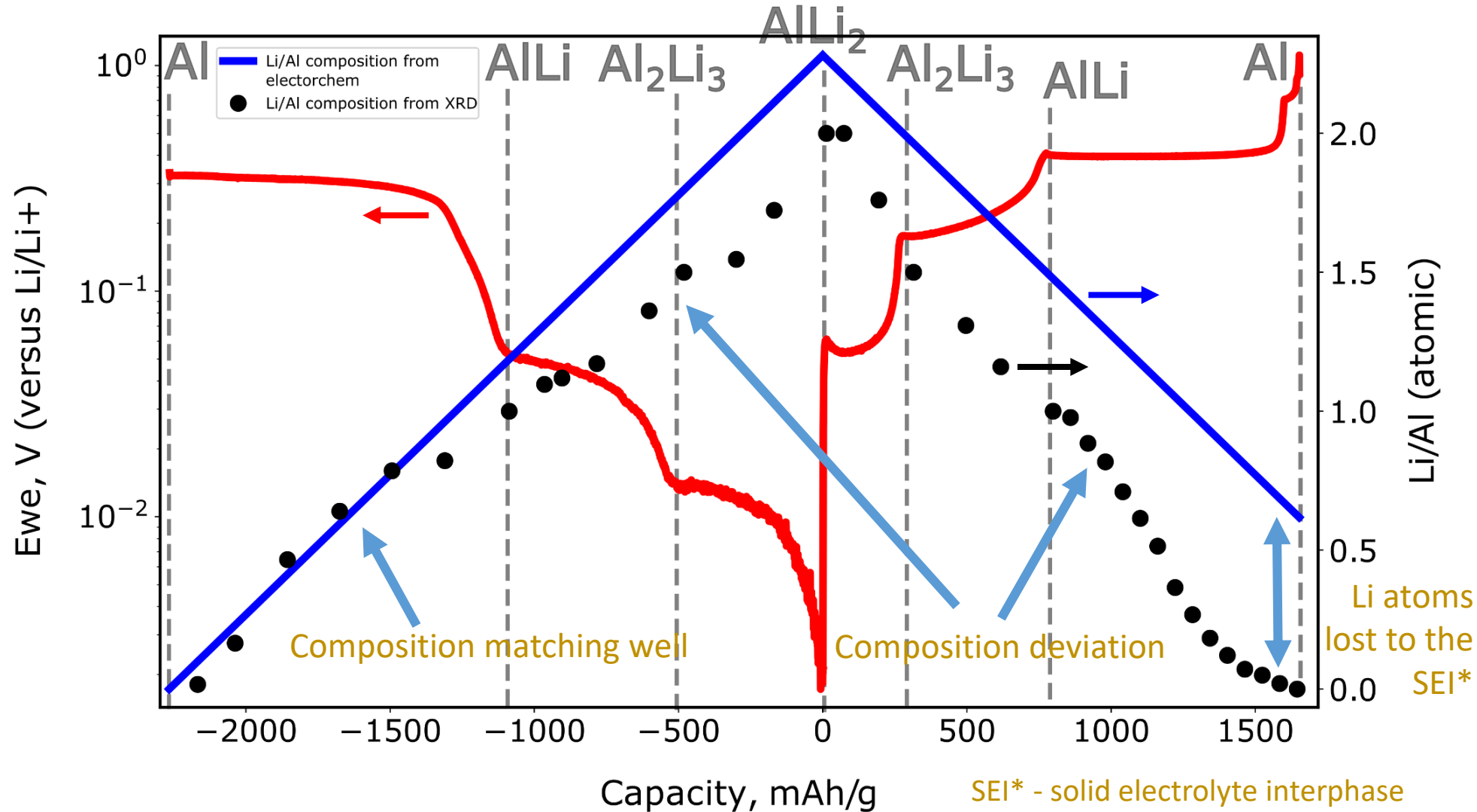
Multiphase refinement of Al and  $\beta$ -AlLi mixture.



# Electrode composition

Blue line – composition calculated from the cell current.

Black dots – composition obtained from structure refinements.



# 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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