Battery activities at AU: Hydrothermal synthesis of nanoparticles

First DBS meeting

March 1st 2013

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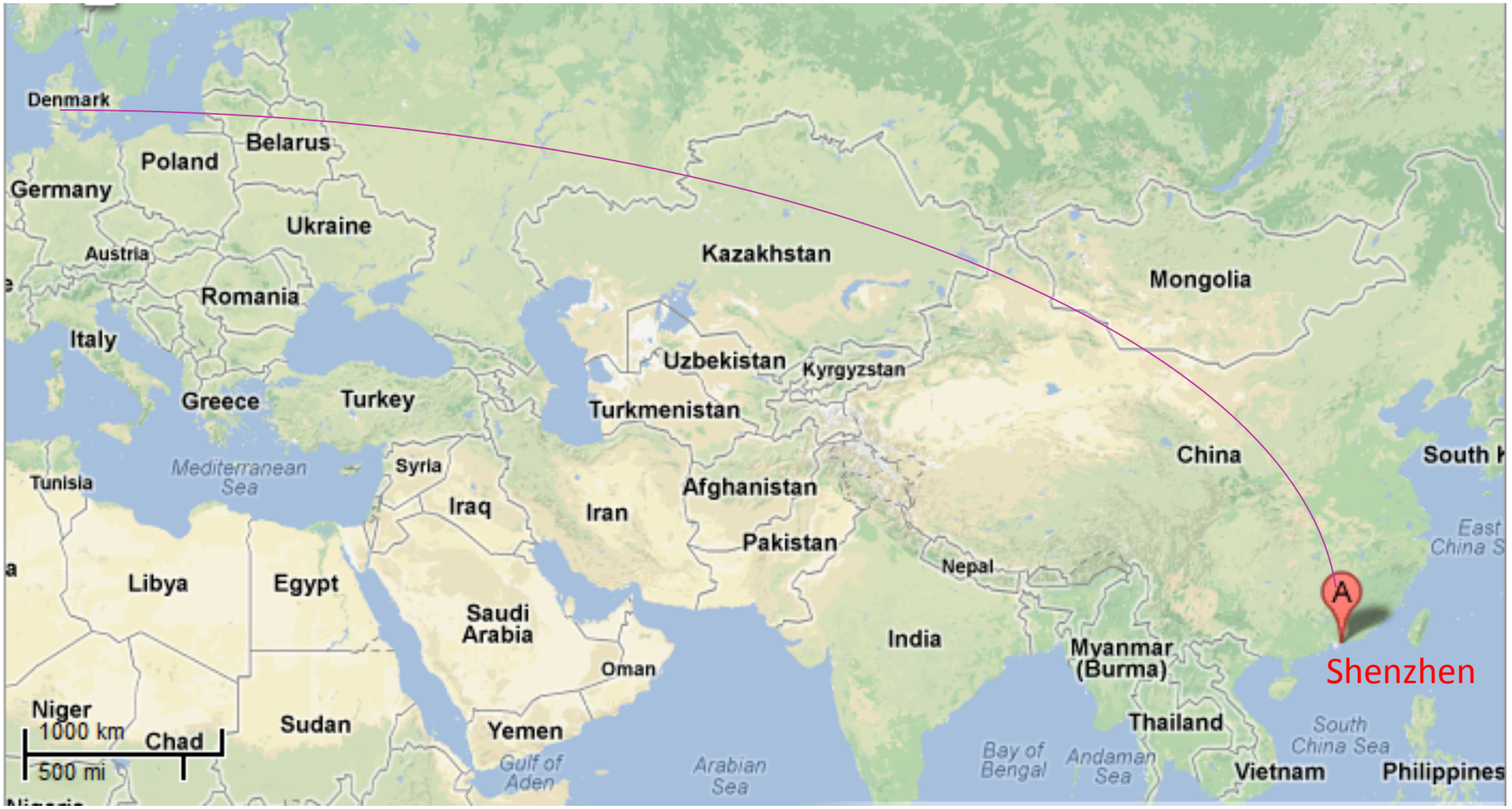
Aarhus University

The story about how I came to Denmark



The story about how I came to Denmark

Outline

- The AU battery people and focus
- Battery facilities at AU
- One step synthesis of $Li_4Ti_5O_{12}$ nanocrystals in a pulse flow reactor
- Conclusion and outlook

The AU battery people and focus

Bo Brummerstedt Iversen

The AU battery people and focus

2008, *In situ* studies of hydrothermel rea

Kirsten Marie Ø. Jensen

Andreas Laumann

2009-2009, hydrothermel flow synthesis

Laumann et al., J. Electrochem. Soc. 2012, 159, A166

Yanbin Shen

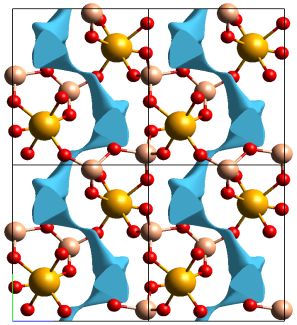
2010, hydrothermel synthesis, *in situ* PXR

Supti Das

2013

Steinar Birgisson

2013, *In situ* studies of hydrothermal reaction



Mette S. Filsø

2011, PED Void Space calculations

Mapping possible Li migration pathways

2013, TiO_2 Martin Søndergaard

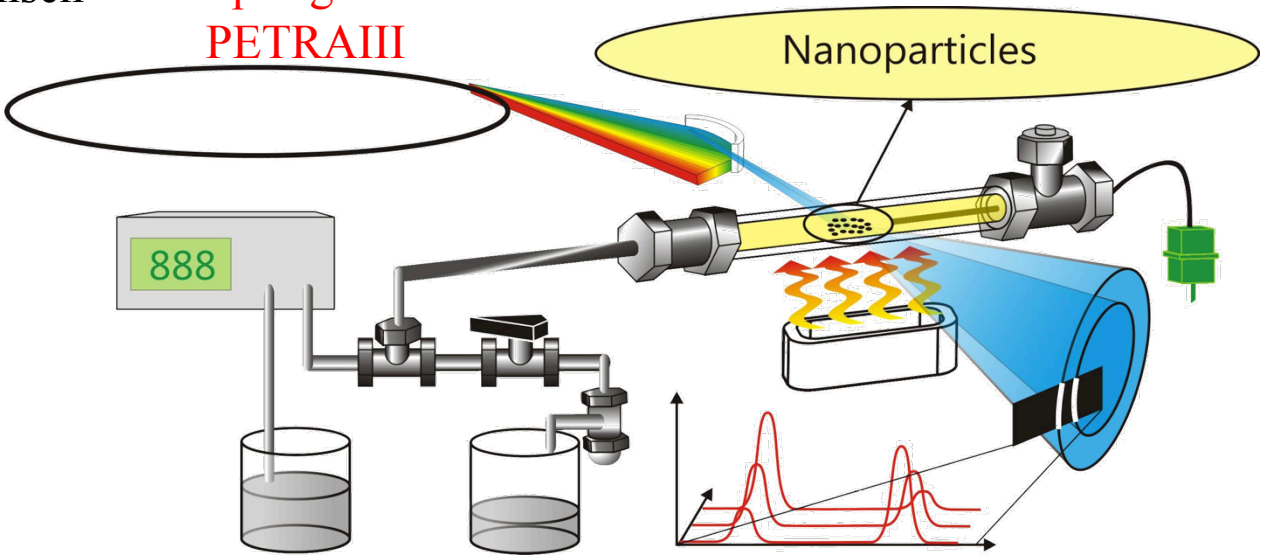
The AU battery people and focus

2008: *In situ* studies of hydrothermel reactions by XRD. Characterization of the materials by synchrotron radiation as they form.

Kirsten Marie Ø. Jensen

MAX-lab
ESRF
APS
Spring-8
PETRAIII

$LiCoO_2$
$LiFePO_4$
SnO_2
.....



Jensen et al., Crystal Growth & Design **2011**, *11*, 753–758
Jensen et al., *J. App. Cryst.* **2011**, 44, 287-294
Jensen et al., *J. Am. Chem. Soc*. **2012**, 134, 6785–6792

PXRD, Rietveld refinement
Total scattering measurements
Pair distribution function analysis

Battery facilities at AU

- Synthesis and characterization of electrode materials
- Moisture controlled battery lab and test station for battery assembly and analysis
- *In-situ* battery setup for *in-situ* PXRD studies on battery while charging/discharging

AU battery facilities - materials

Synthesis

- A lot of Furnaces
- Autoclave
- Flow reactor
- Pulse flow reactor
- Ball mill

Structural characterization

- X-ray diffractometers
- Partners at synchrotron and neutron facilities
- SEM & TEM

Additional

- ICP
- XRF
- XPS
- BET
- TGA/DSC

AU battery facilities - Battery lab

Mixer

Coater

Compressor

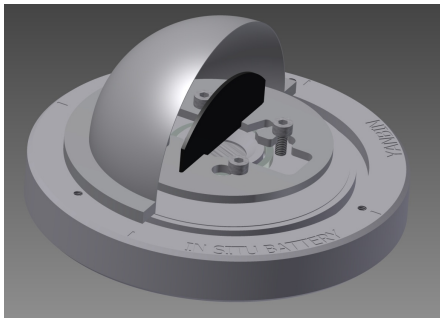
Coin cell

Split cell

Cuter

Vacuum furnace

Crimping machine



In situ cell

AU battery facilities

--Electrochemical performance measurement

Battery analyzer

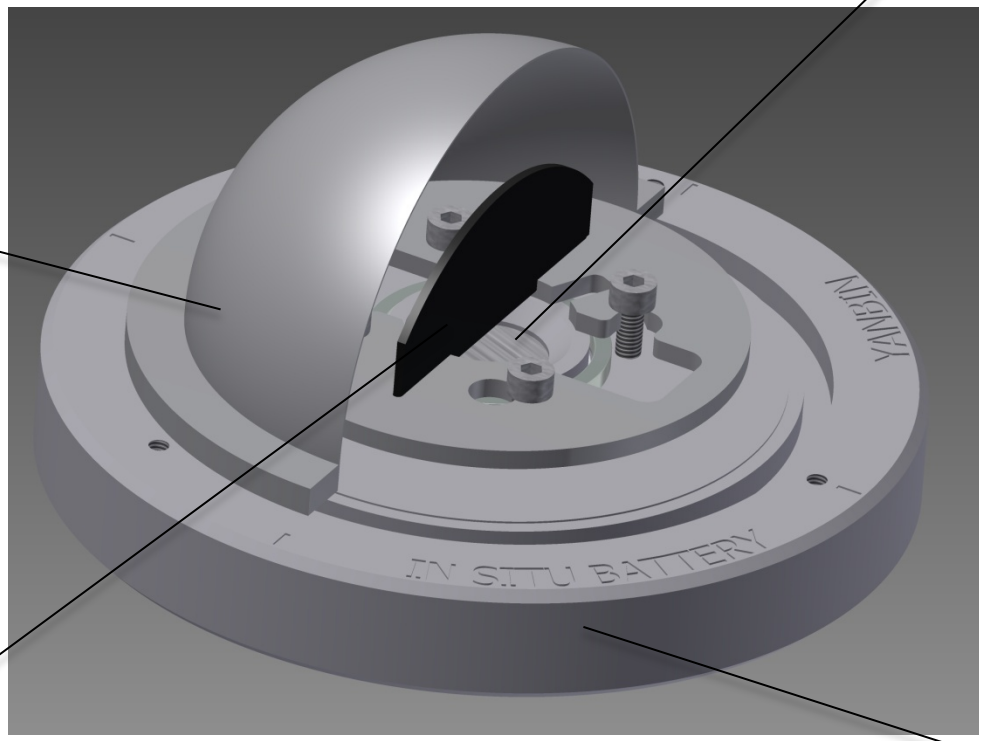
- Voltage/capacity
- Rate-ability
- High/low temperature discharge
- Cycle life

Impedance analyzer
Group of Organic Surface Chemistry

- Electrochemical impedance spectroscopy (EIS)
- Cyclic Voltammetry (CV)
- ....

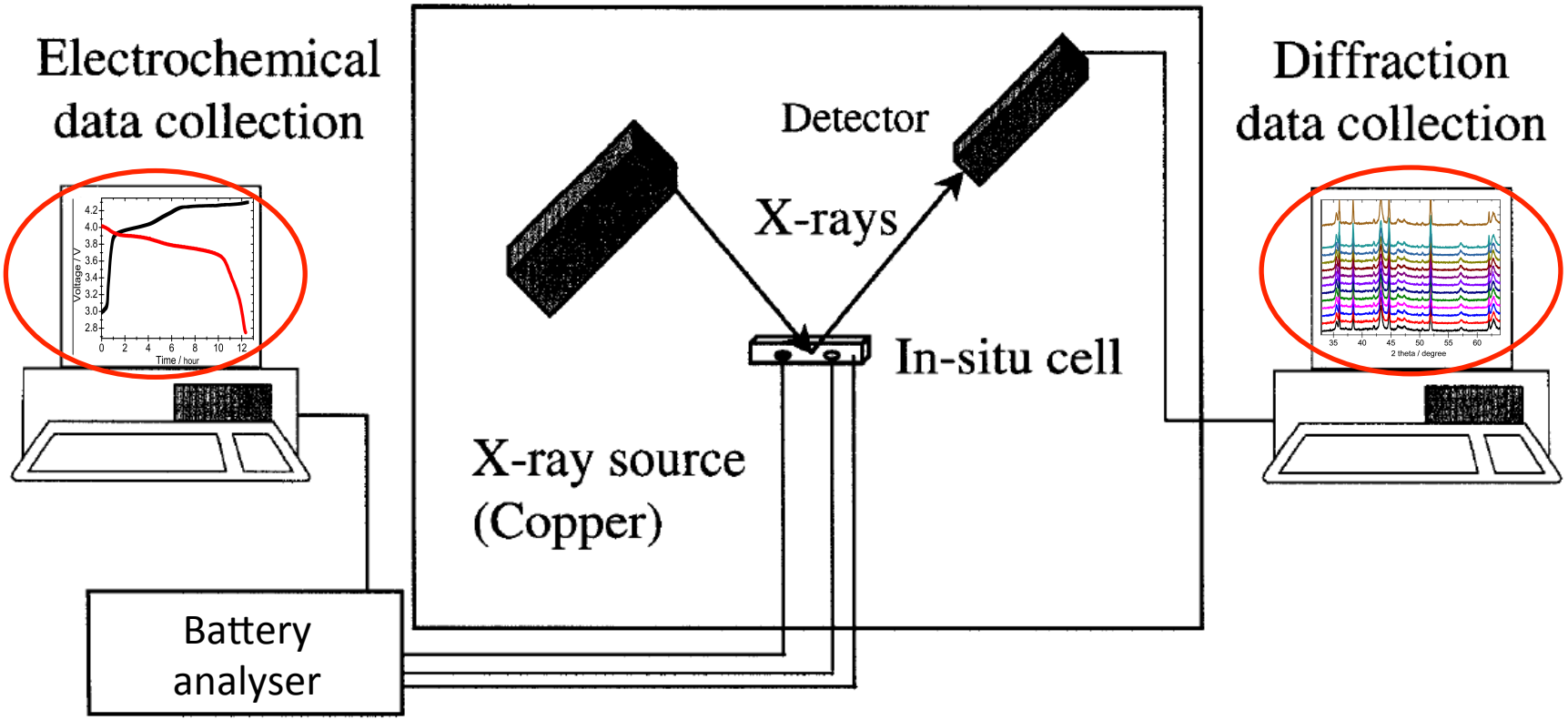
AU battery facilities

----In situ XRD battery



In situ XRD while charge/discharge

Slow charge-discharge (0.05C) and fast X-ray scan (~12 min- lab source)

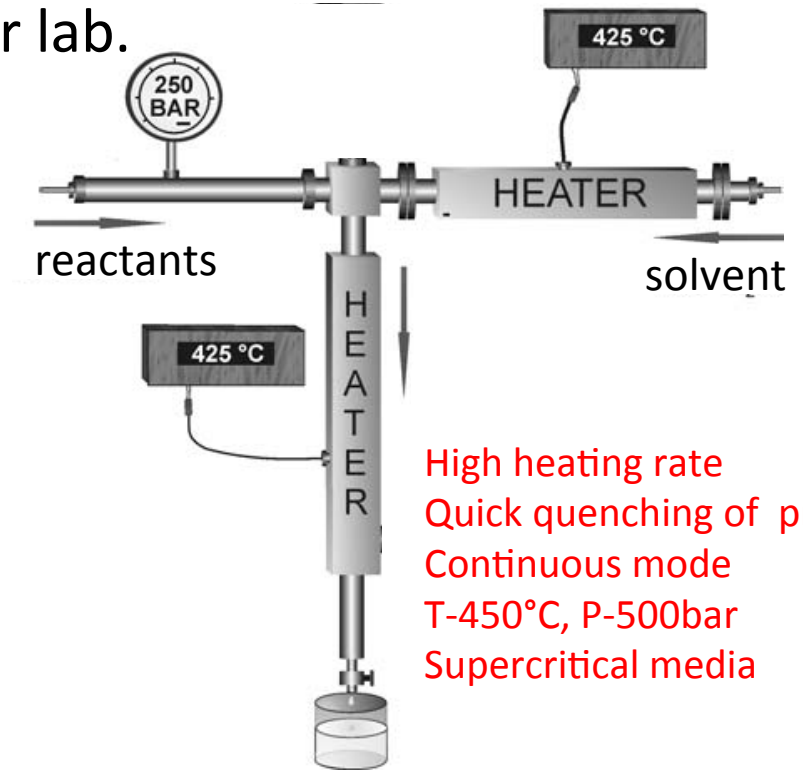


Experimental setup for in-situ battery data collection

Hydrothermal synthesis of nanoparticles

- Hydrothermal and solvothermal synthesis is excellent method for preparing crystalline nanoparticles
- Various particle sizes, crystallinities and morphologies can be obtained by adjustment temperature, pressure, concentration, pH value etc.
- It is standard technique in our lab.

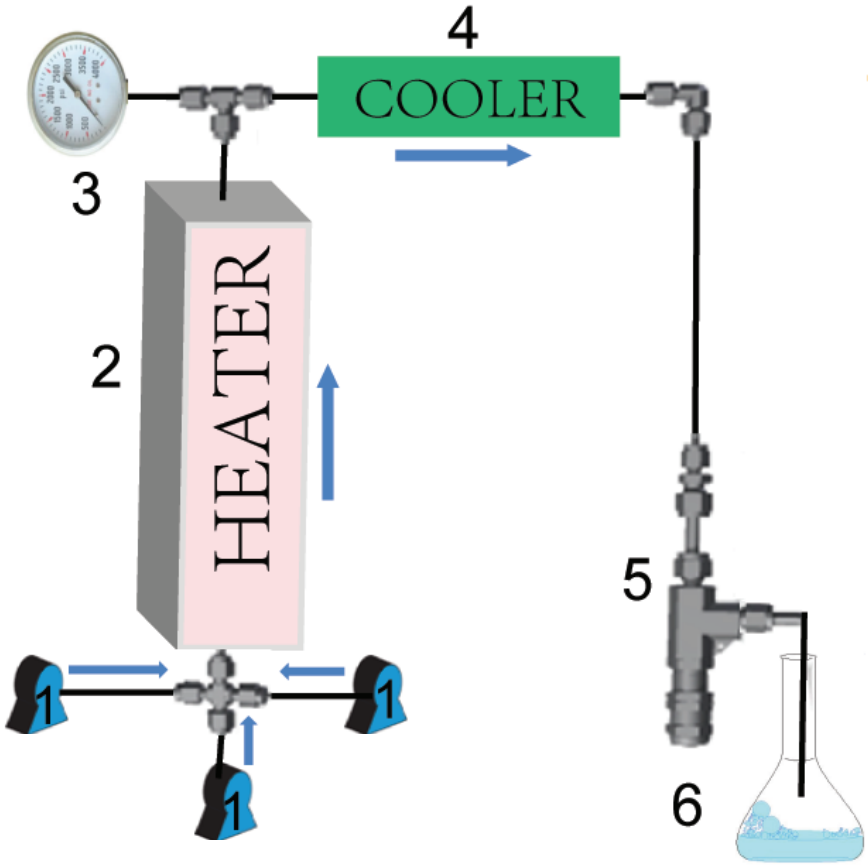
batch autoclave reactors
Simple, easy to use



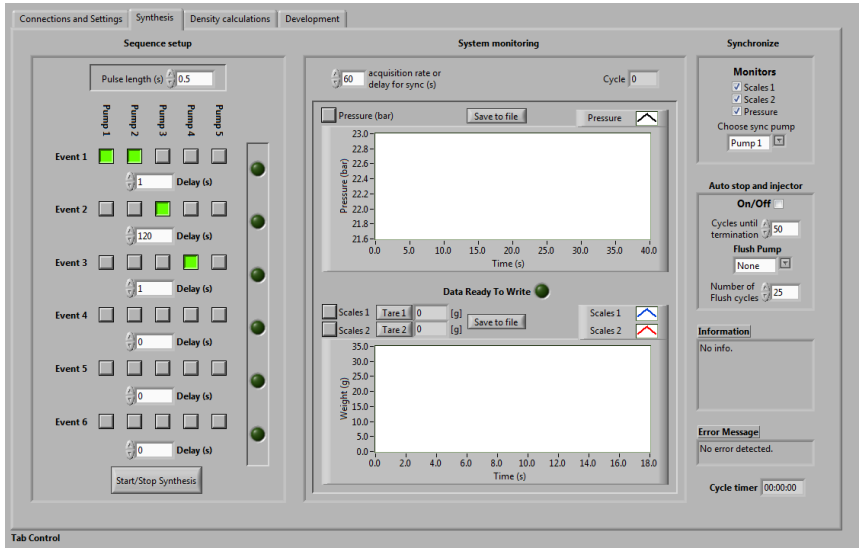
High heating rate
Quick quenching of product
Continuous mode
T-450°C, P-500bar
Supercritical media

Flow reactor

A pulse flow reactor



The electronic control box

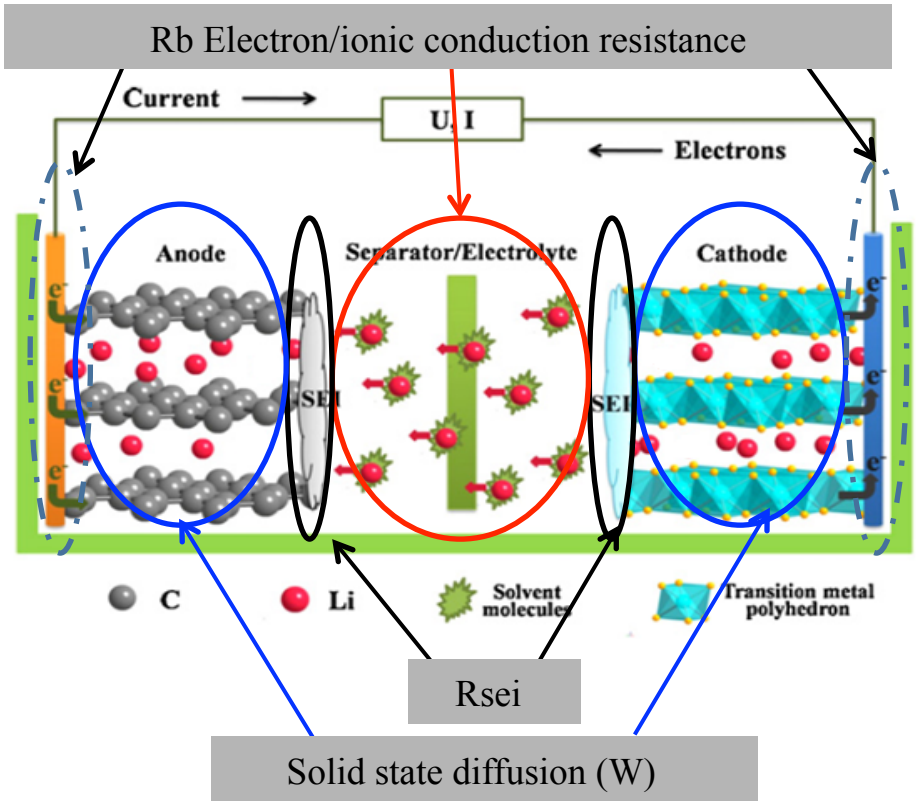


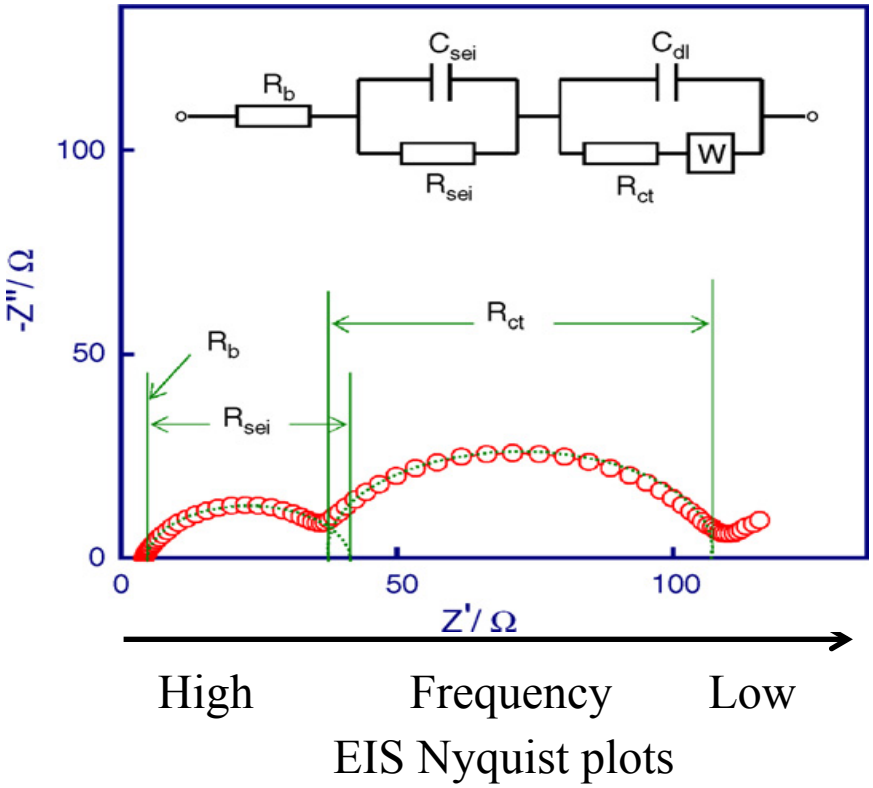
Developed by Jakob Rostgaard Eltzholtz

Electrochemical impedance Spectroscopy (EIS)

AC voltage - 5 mV amplitude on cell

+ Charge transfer - Rct





High Frequency Low

EIS Nyquist plots

Rb- Resistance of the electrolyte, separator - The intercept on the Z-real axis

Rsei –Resistance of the SEI -Semicircle in the high frequency range

Rct-Charge transfer resistance at the particle/electrolyte interface - Semicircle in the middle frequency range

W- Warburg impedance for lithium diffusion -The sloping line at low frequencies

Csei-Capacitance of the SEI on the surfaces of graphite

Cdl- double-layer capacitance

Conclusions

- **In Aarhus, we focus on:**
  - Hydrothermal synthesis of nanoparticles for batteries
  - *In situ* X-ray studies of hydrothermal synthesis
  - *In situ* PXRD studies of charging/discharging
  - Crystallographic studies of battery materials
  - PED void space calculations for mapping of possible Li diffusion pathways
- **We have most of the conventional facilities for:**
  - Material synthesis
  - Material characterization
  - Battery lab
- **Continuous Pulse flow synthesis** is good method for obtaining hig h

  purity $Li_4Ti_5O_{12}$ nanoparticles with various sizes and crystallinities.

Outlooks

- In-situ hydrothermal X-ray studies on more battery materials wit h various techniques (PXRD,SAXS, total scattering, PDF…)
- In situ XRD study on the material structure while charging and discharging
- Develop more advanced hydrothermal synthesis reactor for new structure material (e.g. core/shell) synthesis
- Develop more cheap, green methods for synthesis of electrode material
- We are open to collaboration from all of you

Acknowledgements

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The battery group

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Steinar Birgisson

Supti Das

Mogens Christensen

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Founding

Questions or comments?