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Workshop

Electrode Materials for Sodium-Ion Batteries II.

TU Wien
3. May 2019

TECHNISCHE UNIVERSITÄT WIEN

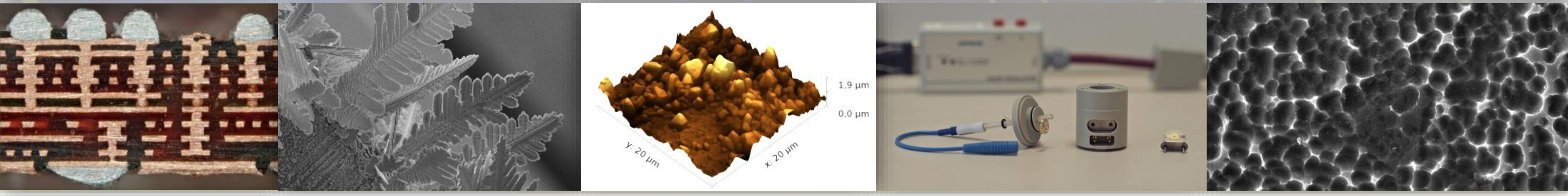
Dr. Jiri Libich

FACULTY OF ELECTRICAL ENGINEERING AND COMMUNICATION
department of electrical and electronic technology

Centre for Research and Utilization of Renewable Energy

Presentation outline

- Brno, Czech Republic
- Brno University of Technology
- Department Profile (Department of Electrical and Electronic Technology)
- Our work relate with electrochemical power sources
- Research field – Sodium-ion batteries
- Conclusion



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Brno University of Technology

- Established 1899
- Over 24 000 students
- Divided to 8 faculties and 2 institutes

BRNO UNIVERSITY OF TECHNOLOGY

CEITEC	Central European Institute of Technology BUT	+
FA	Faculty of Architecture	+
FBM	Faculty of Business and Management	+
FCE	Faculty of Civil Engineering	+
FEEC	Faculty of Electrical Engineering and Communication	+
FFA	Faculty of Fine Arts	+
FCH	Faculty of Chemistry	+
FIT	Faculty of Information Technology	+
FME	Faculty of Mechanical Engineering	+
IFE	Institute of Forensic Engineering	+

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Faculty of Electrical Engineering and Communication (FEEC)

- Over 3500 students
- Divided to 14 departments

BRNO UNIVERSITY OF TECHNOLOGY FACULTY OF ELECTRICAL ENGINEERING AND COMMUNICATION

- Our department : Department of Electrical and Electronic Technology (UETE)

FACULTY OF ELECTRICAL ENGINEERING AND COMMUNICATION department of electrical and electronic technology

1. Electrochemical power sources (batteries)
2. Renewable energy (photovoltaics, wind power)
3. 3D modeling and simulation
4. Dielectric materials and isolants
5. Technology od PCB, design, interconnection structures

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Profile of Department of Electrical and Electronic Technology (UETE)

• Lithium, lead acid, redox flow batteries, fuel-cells • Post-Lithium systems (Li-Sulphur, Na-Ion) • Photovoltaic (FV)	Energy sources
• Diagnostics of material properties – XRD • Climate tests • Corrosion Tests • Surface Diagnostics – ESEM, AFM	Materials
• Diagnostics of the soldering process, technology • Surface treatment	Technology
• 3D Modeling and Simulations • Design of electronics and firmware	Simulations and computer design

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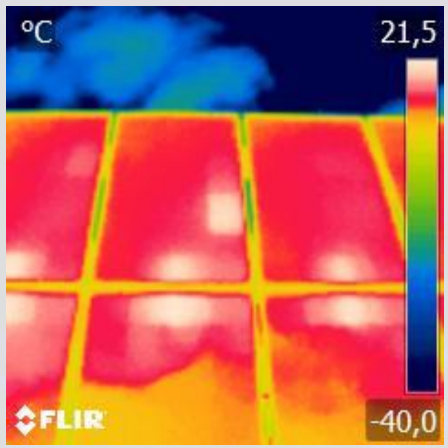
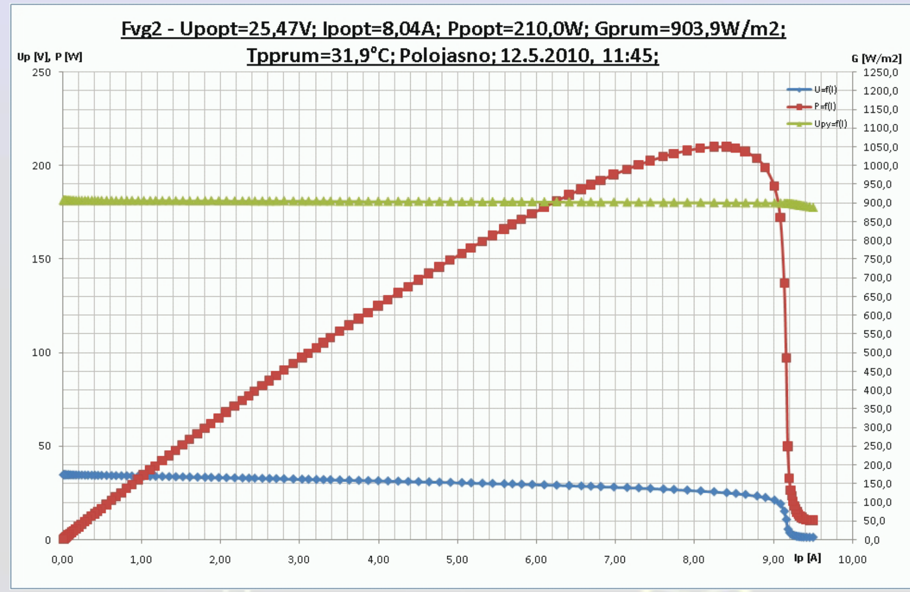
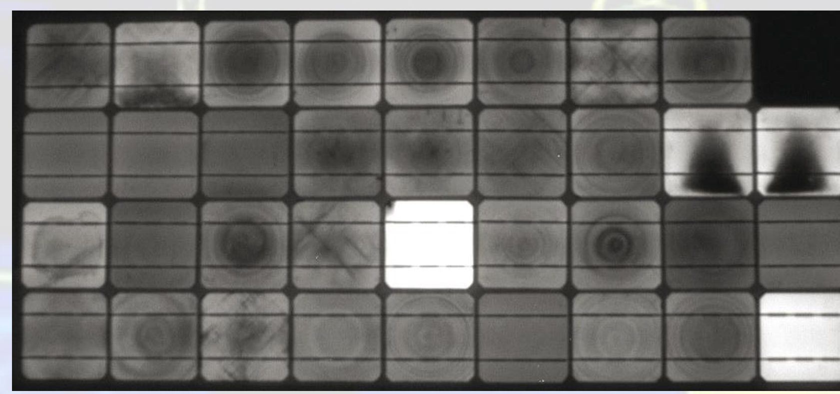
Enegy sources – Renewable

- Diagnostic of photovoltaic panels with help of PASAN instrument
    - Efficiency testing
    - Localization of defects by method electroluminescence







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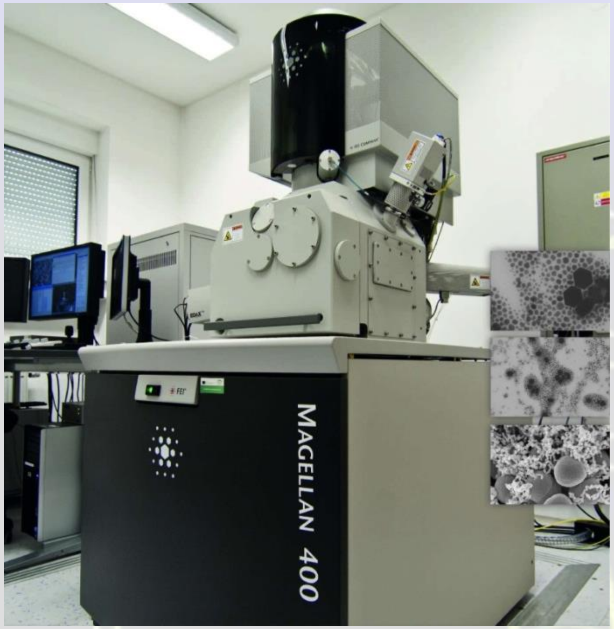
Materials

- Instruments: ESEM, AFM, XRD



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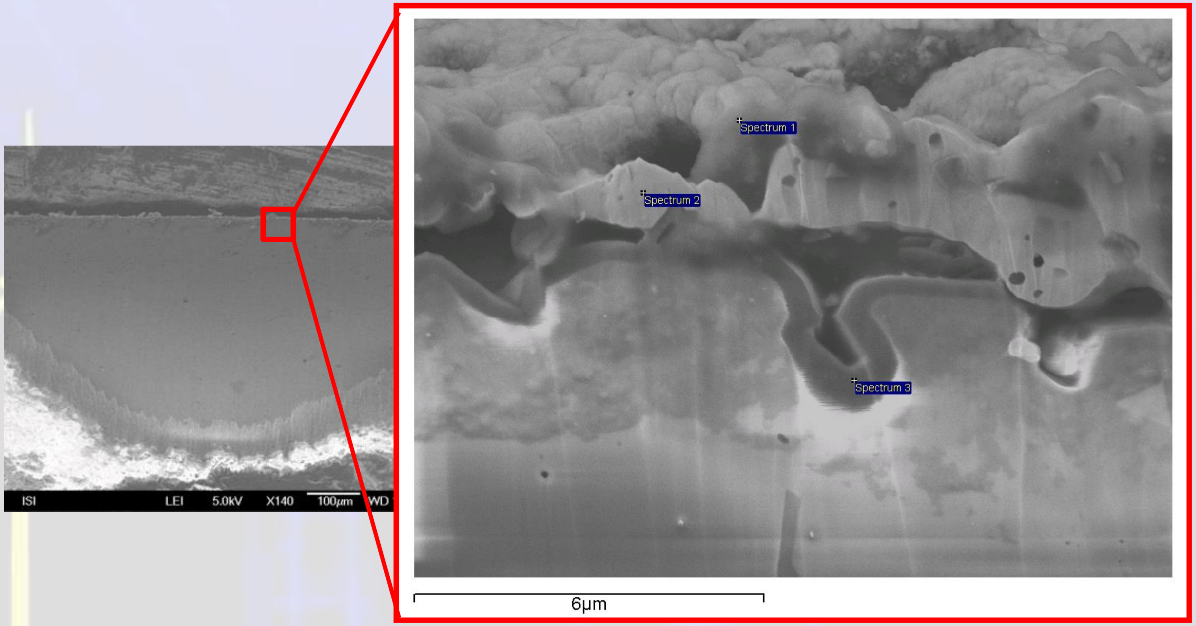

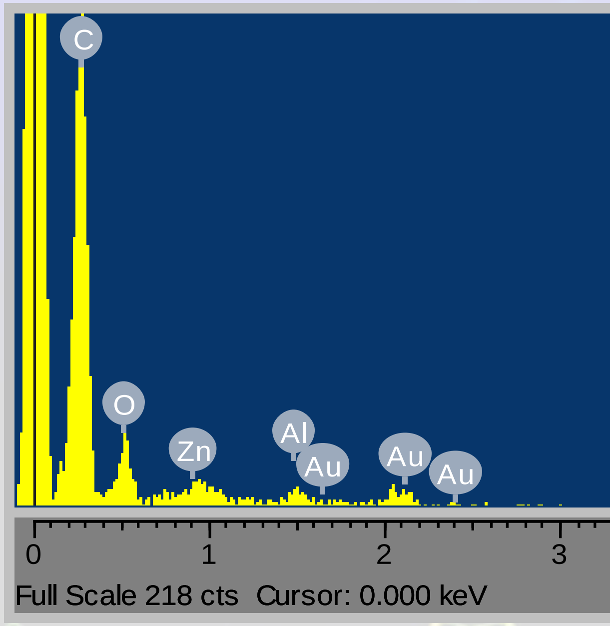

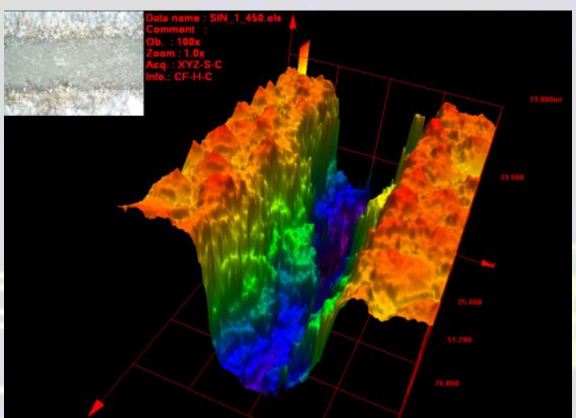

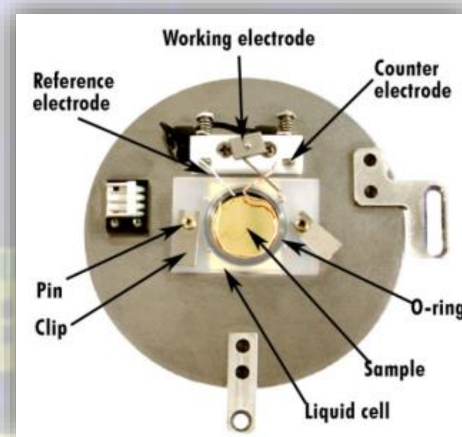

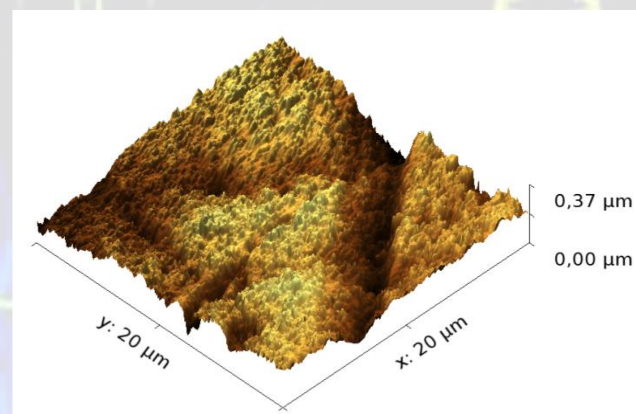

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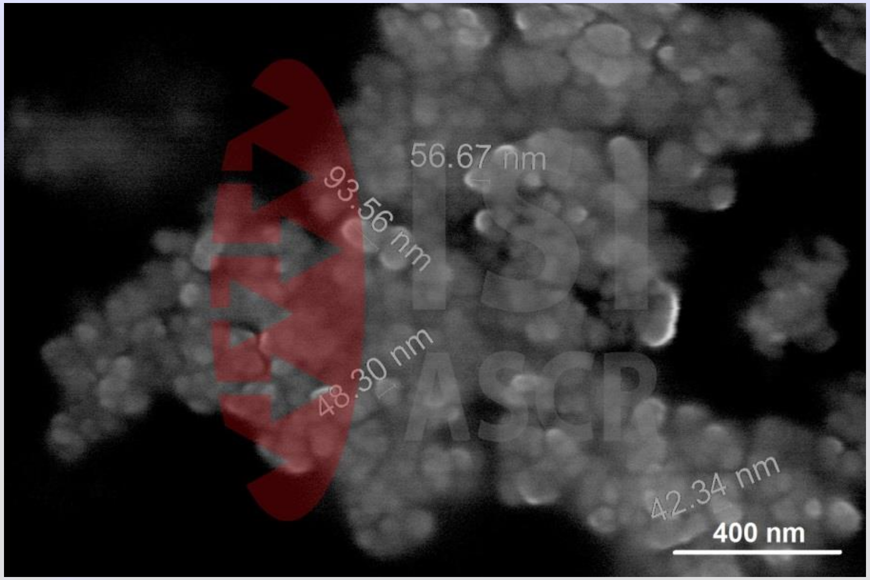

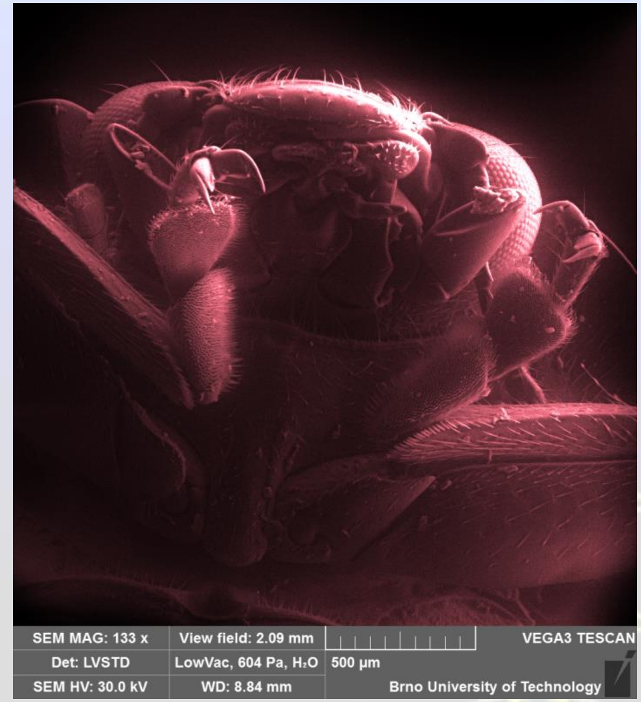

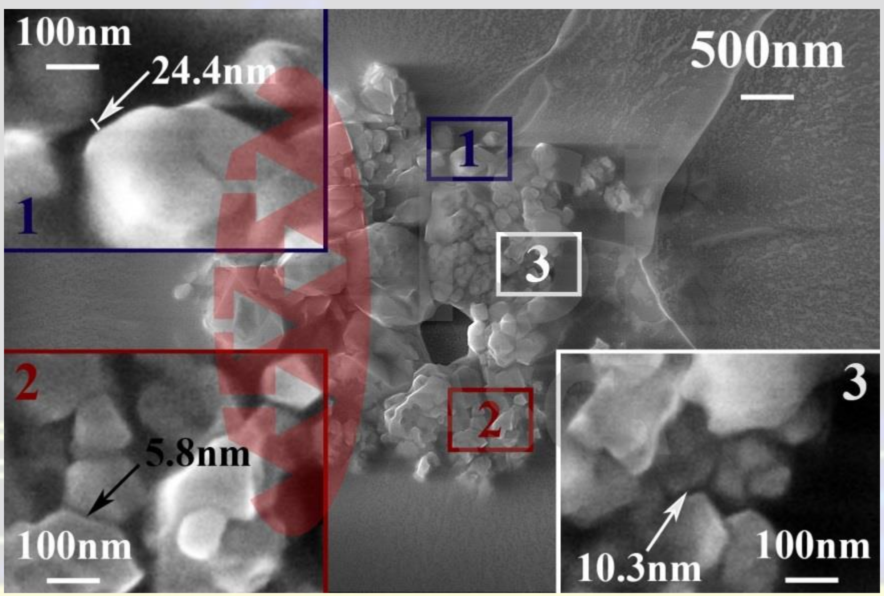

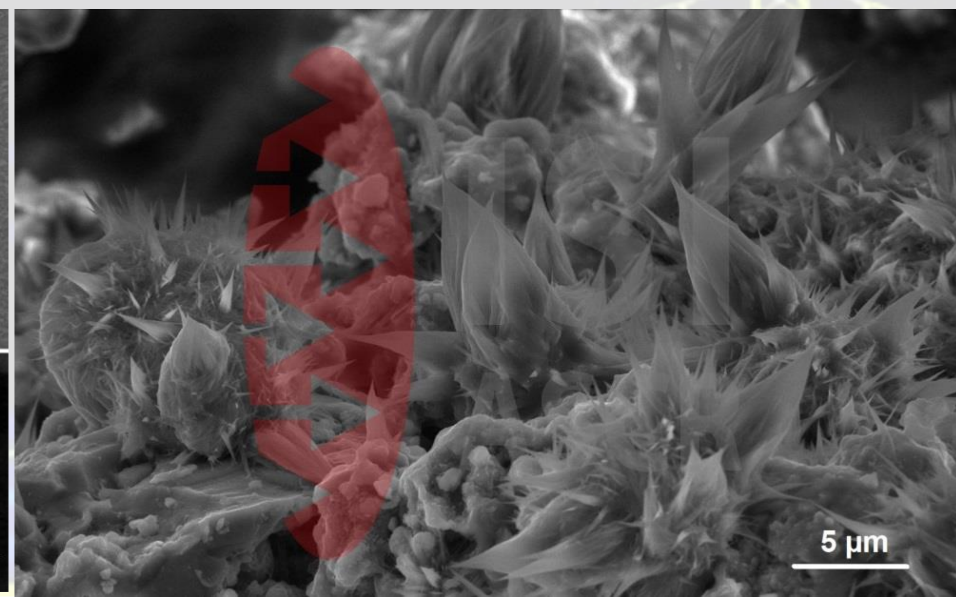

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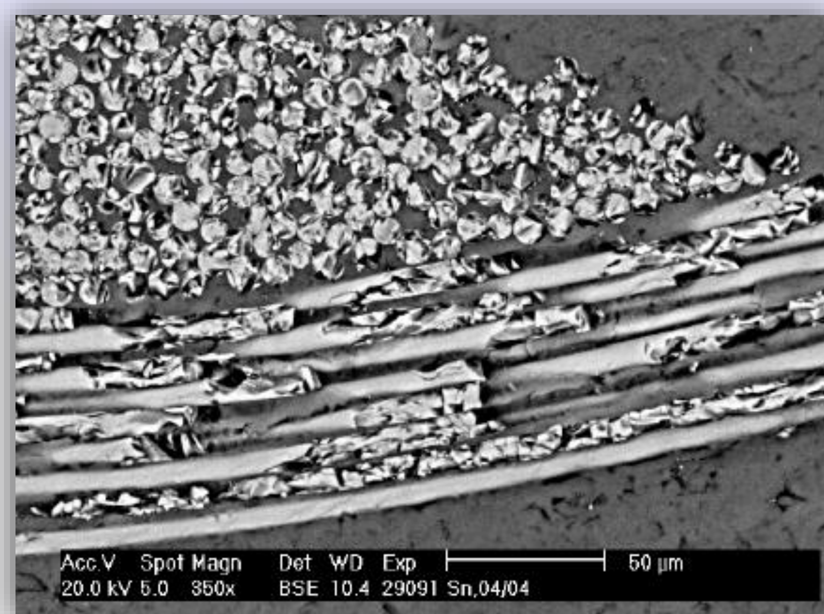
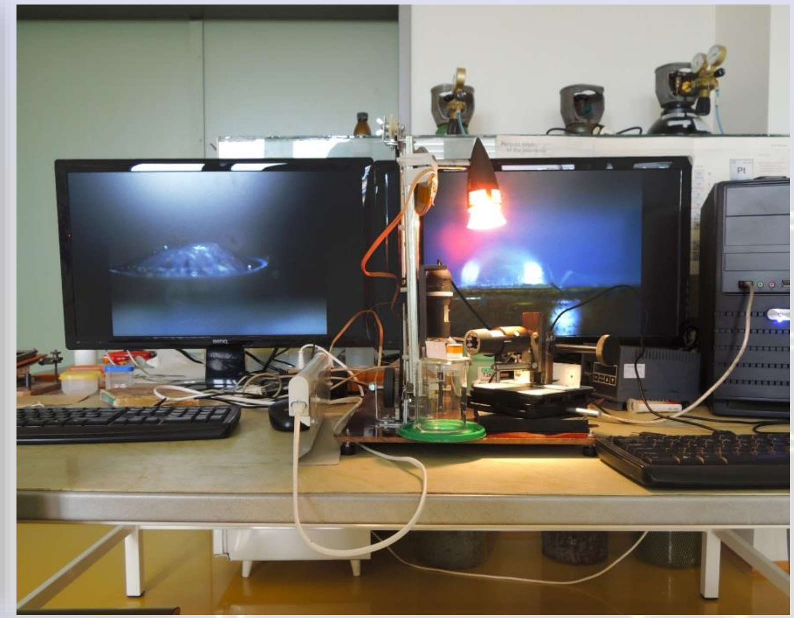
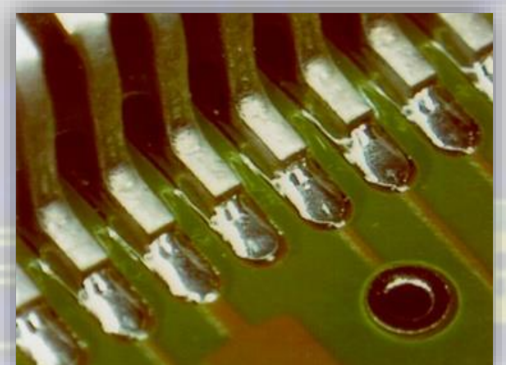
Technology

- Assembly and interconnection technologies, defect analysis







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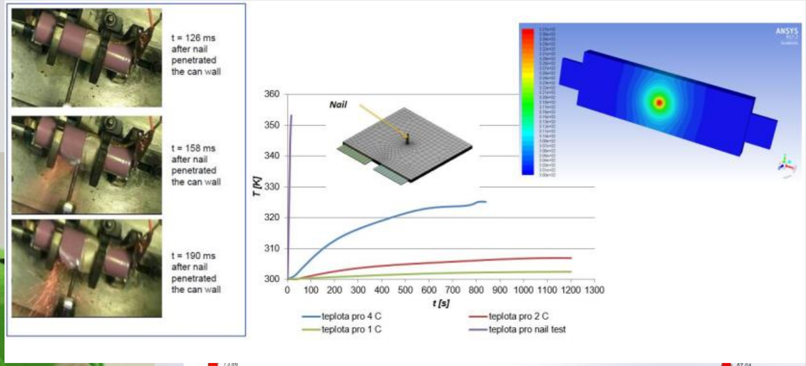
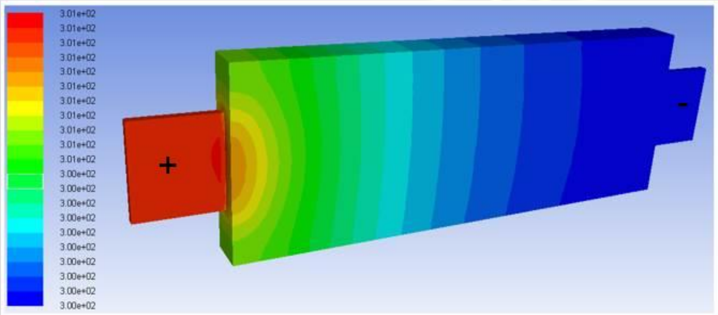
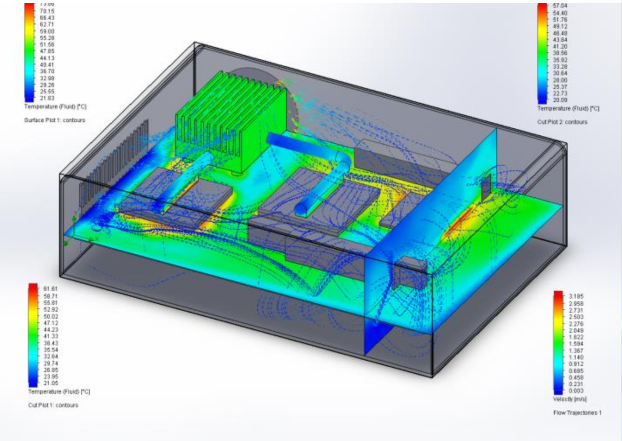
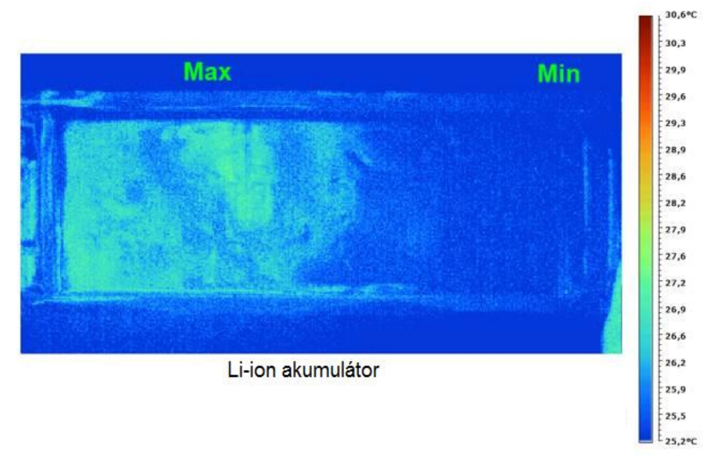
Simulation and 3D computer design

- SolidWorks, Ansys
- In systems using finite volumes and elements
- Designing a numerical model to create a uniform flow under all standardized conditions.
- Creation of a numerical model of ultrasound beam propagation.
- Analysis of the influence of climatic conditions on the gas meter prototype (heat transfer).
- Simulation of electric strength, electric fields and antistatic prevention on a prototype.









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Enegy sources – Electrochemistry

- University has a long tradition of research in energy storage application

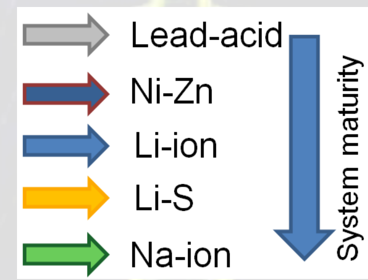
1966 lead-acid

1970s Ni-Cd

1979 Ni-Zn

2005 Li-ion

2017



- Selected research activities in terms of electrochemistry
  - Li-ion battery
  - Lithium ion battery for the smart textiles applications
- Post-Li battery systems (Na-Ion, Li-Sulphur)
- Lead-acid batteries
- High-temperature ceramic materials
- Flow-through redox systems

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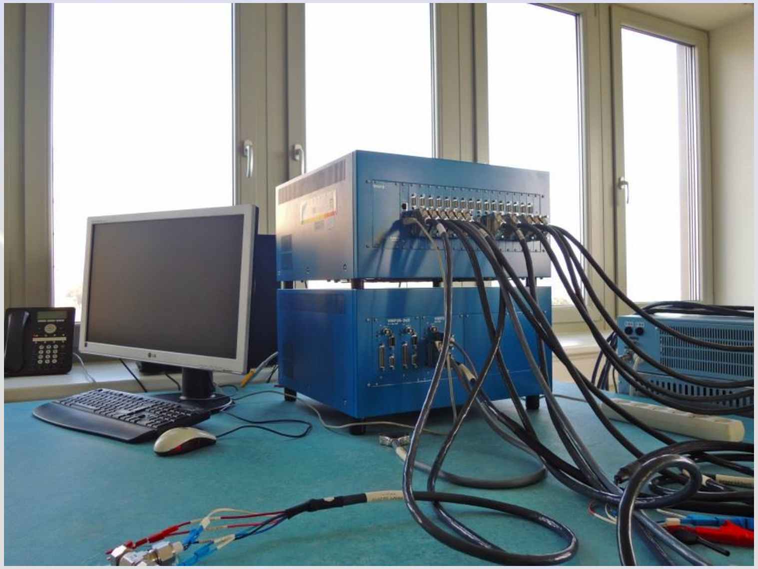
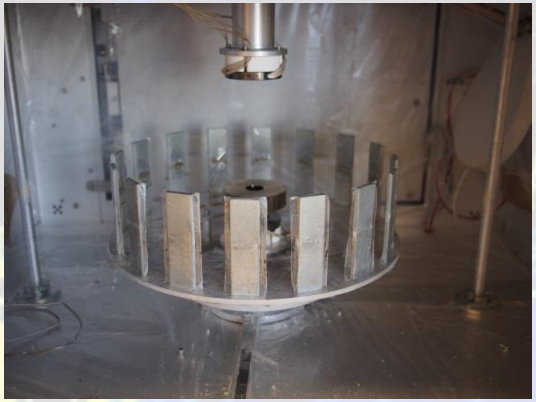
Enegy sources – Electrochemistry

- Equipments: Potentiostat , Gloveboxes, Sputtering device, Forcespinning…





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Enegy sources – Electrochemistry

Current research in electrochemical energy storage systems

- Lithium-Ion Batteries and Post-Lithium systems
  - Basic research of conventional and advanced (5 Volts) $LiFePO_4$, $LiCoO_2$ and $LiMn_2O_4$ batteries in relation to their function, stability and safety.
  - Study of electrolytes for Li-ion batteries – stability at high voltage, flammability,...
  - Development of Li – sulfur system
  - Preparation of complete Li-ion cells + degradation tests
  - Development of Na – Ion system
- Advanced and Alternative Systems
  - Application-oriented research focused on Pb-A battery – resolving the PCL3 effect
  - Investigation of performance aspects of vanadium redox flow with focus on electrode degradation and general vanadium redox kinetics.
  - Continue the development of sodium systems
- Supporting Activities
  - Development of the equivalent electrical circuit models for the studied structures and analytical models describing the aging structures.

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Enegy sources – Electrochemistry

Cathode material for lithium ion accumulators prepared by screen printing for the smart textiles applications

- $LiFePO_4$ based cathode electrode for printed secondary lithium based cells.
- An ink formulation was developed for the screen printing technique.
- Standard PVDF-based binder and conductive additives were replaced by conductive polymers Advanced and Alternative Systems

Code name	Electrode material	Binder	Conductive content	Deposition technique	Cathode Underlayer
LFP-SuP-Prt	$LiFePO_4$	PVDF	Super P	Screen printing	No
LFP-PEDOT-Prt	$LiFePO_4$	PEDOT:PSS	PEDOT:PSS	Screen printing	No
LFP-PEDOT-Prt-C	$LiFePO_4$	PEDOT:PSS	PEDOT:PSS	Screen printing	Carbon
LFP-SuP-BarCoat	$LiFePO_4$	PVDF	Super P	Spiral Bar Coating	No

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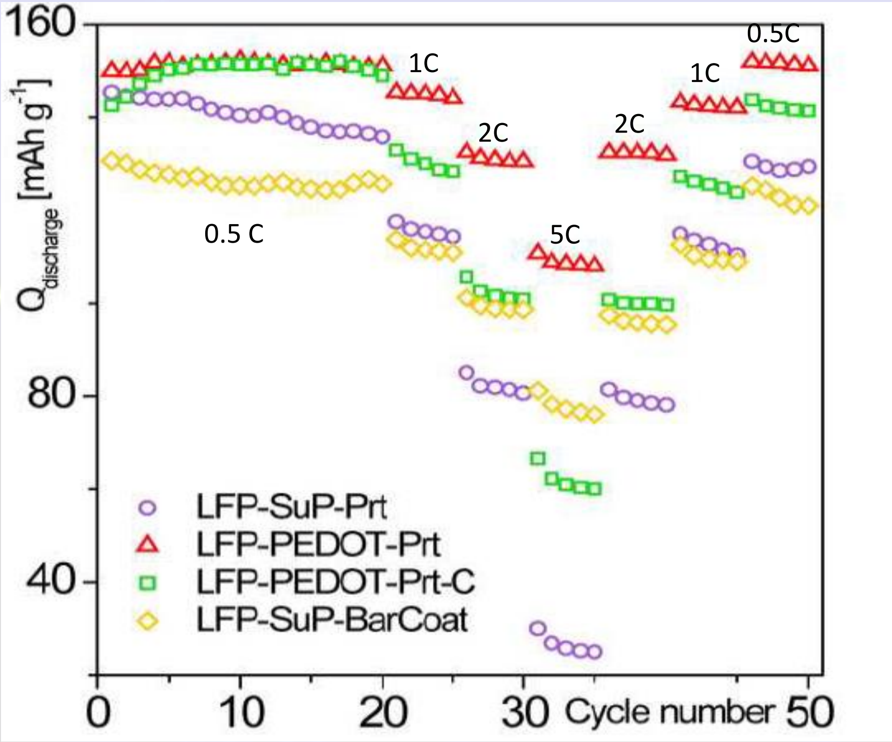
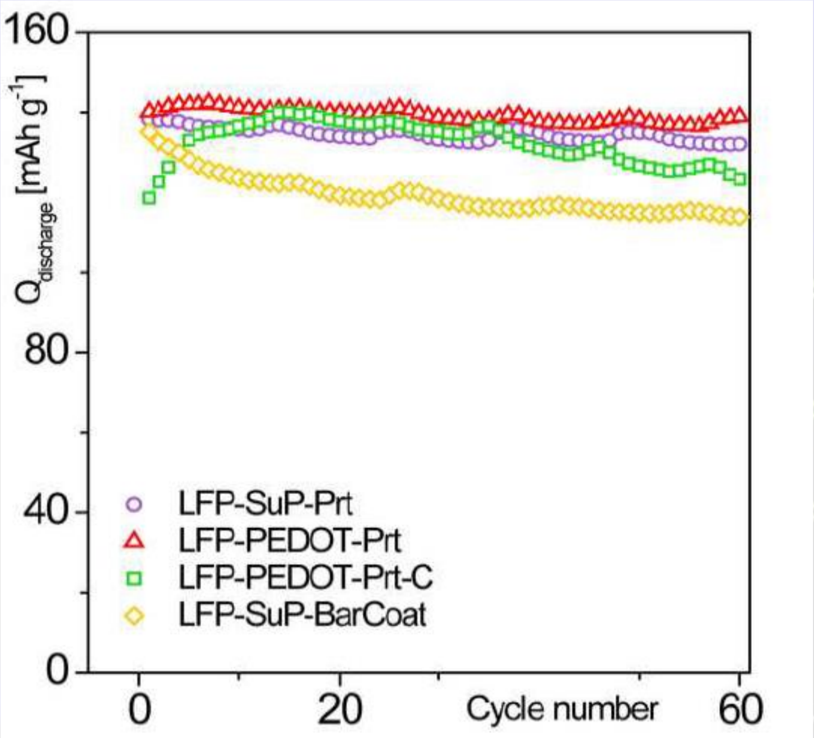
Enegy sources – Electrochemistry



Change of capacity of the $LiFePO_4$ electrode layers for different C-rates: 0.5, 1, 2, 5, 2, 1, and 0.5.



The cycling performance LFP-SuP-Prt, LFP-PEDOT-Prt, LFP-PEDOT-Prt-C, LFP-SuP-BarCoat, at 1 C for 60 cycles.

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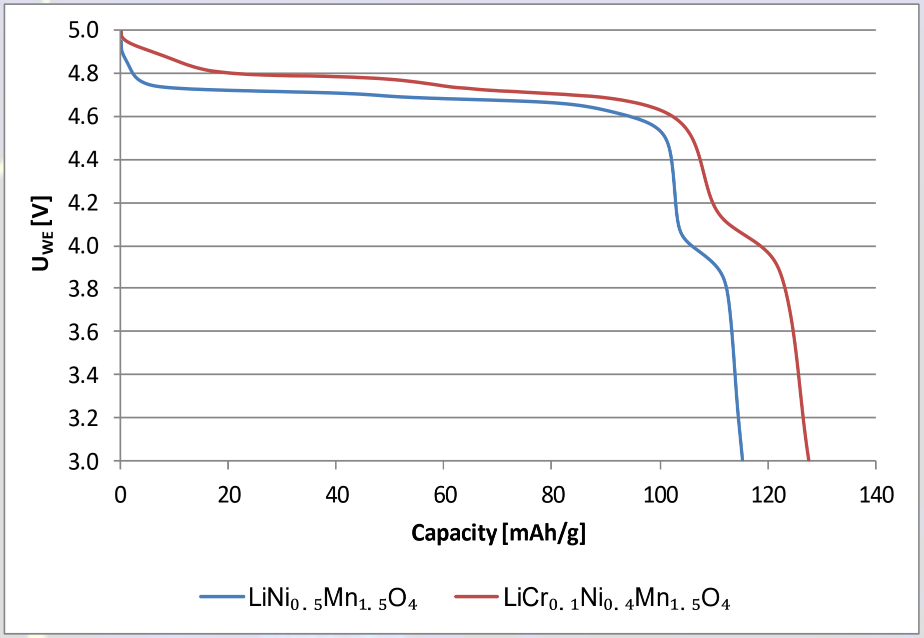
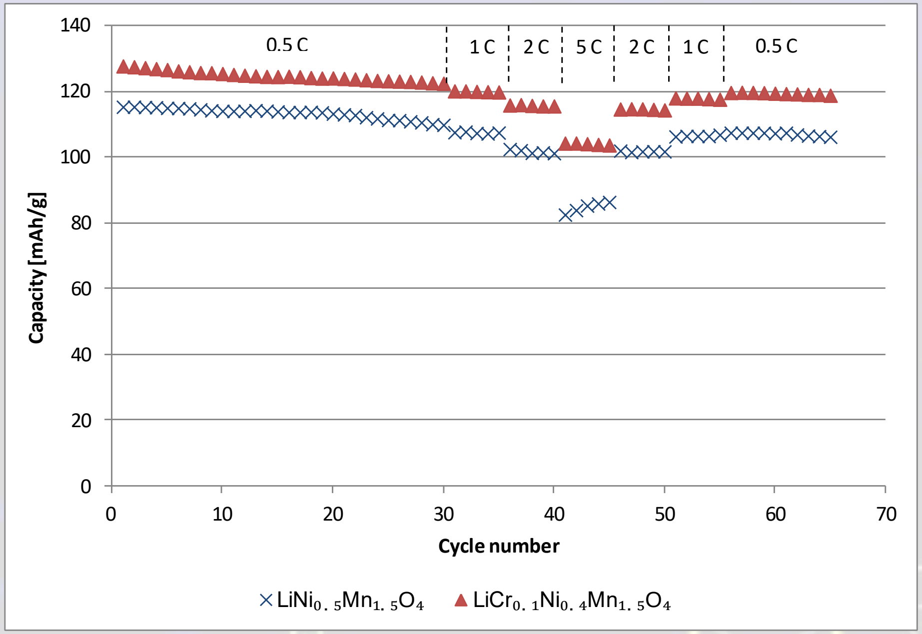
Enegy sources – Electrochemistry

- 5 Volts cathode materials:
  Effect of Cr doping to the properties of LiNi0.5Mn1.5O4



Comparison of first discharge curves of $LiNi_{0.5}Mn_{1.5}O_4$ and $LiCr_{0.1}Ni_{0.5}Mn_{1.5}O_4$ at 0.5 C



Comparison of capacity change depending on the load for materials $LiNi_{0.5}Mn_{1.5}O_4$ and $LiCr_{0.1}Ni_{0.5}Mn_{1.5}O_4$

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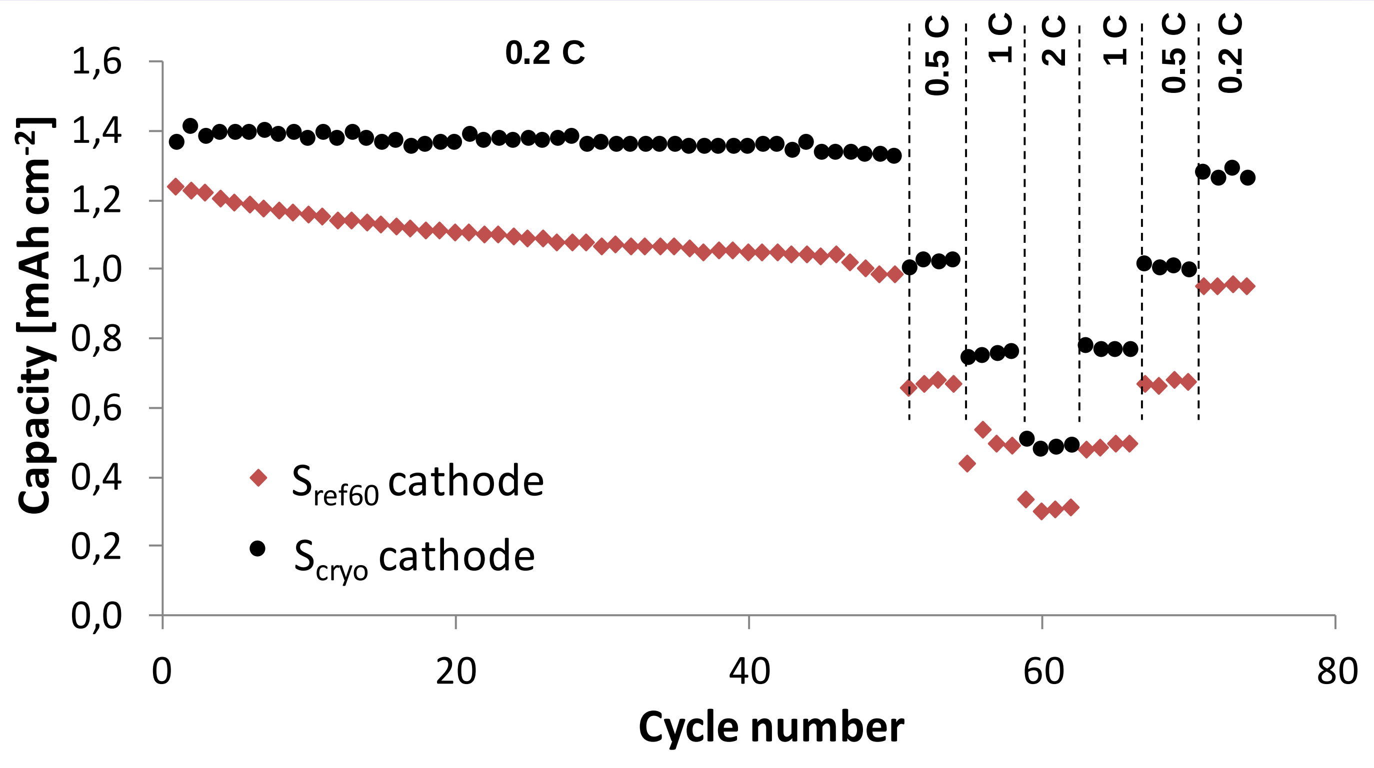
Enegy sources – Electrochemistry

- Li-Sulfur Battery Systems



Changes of capacity of Sref60 cathode and Scryo cathode

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Na-Ion Storage Systems
Enegy sources – Electrochemistry

- Sodium is abundant, 6th most abundant elements in the earth crust (lithium occupies 33th position), it reach year produciton over 225 M tonnes (litihum just around 0.043 M tonnes)

- Sodium-Ion batteries, most promisable system, next generation of electrochemical power sources, batteries for renewable energy harvesting, cheap, environmental friendly…

- Sodium-ion batteries work on the same principles as the well-known and described lithium-ion batteries, they use same technology most of them use aprotic electrolytes DMC (dimethyl carbonate), EC (ethylene carbonate), PC (propylene carbonate) with salts as $NaCLO_4$ (sodium perchlorate) or $NaPF_6$ (sodium hexafluorophosphate)

- Cathode materials for sodium-ion batteries, similar to convention stable cathode materials for lithium-ion batteries. Cathode material $NaCoO_2$ (sodium cobalt oxide) or $NaNi_{1/2}Mn_{1/2}O_2$ (Sodium nickel manganese oxide)

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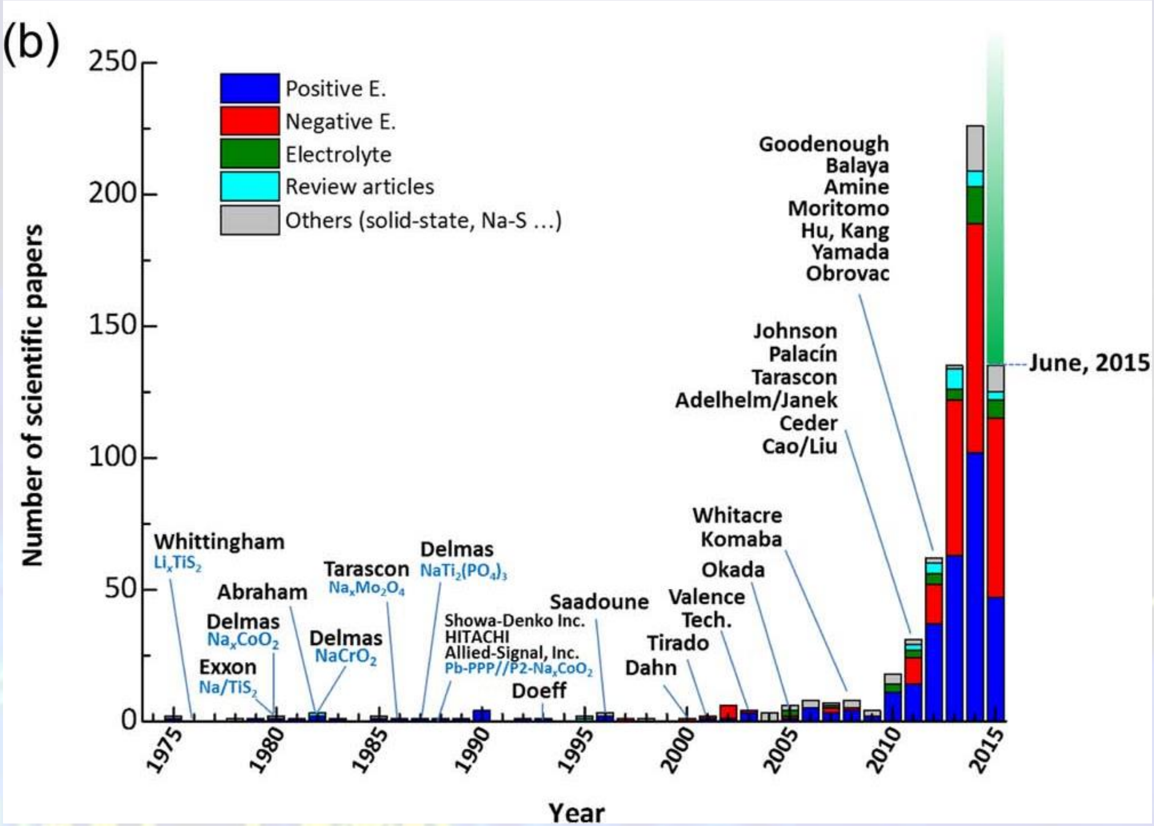
Na-Ion Storage Systems

- Anode materials are issue, Lithium-Ion batteries use manly graphite as active electrode materials for negative electrode (anode), in case of sodium ion it is not possible to use graphite, because sodium ion having large diameter and cannot be inserted among graphite sheets



G. Rousse et .al., Chemistry of Materials 25 (2013) 4946–4956.

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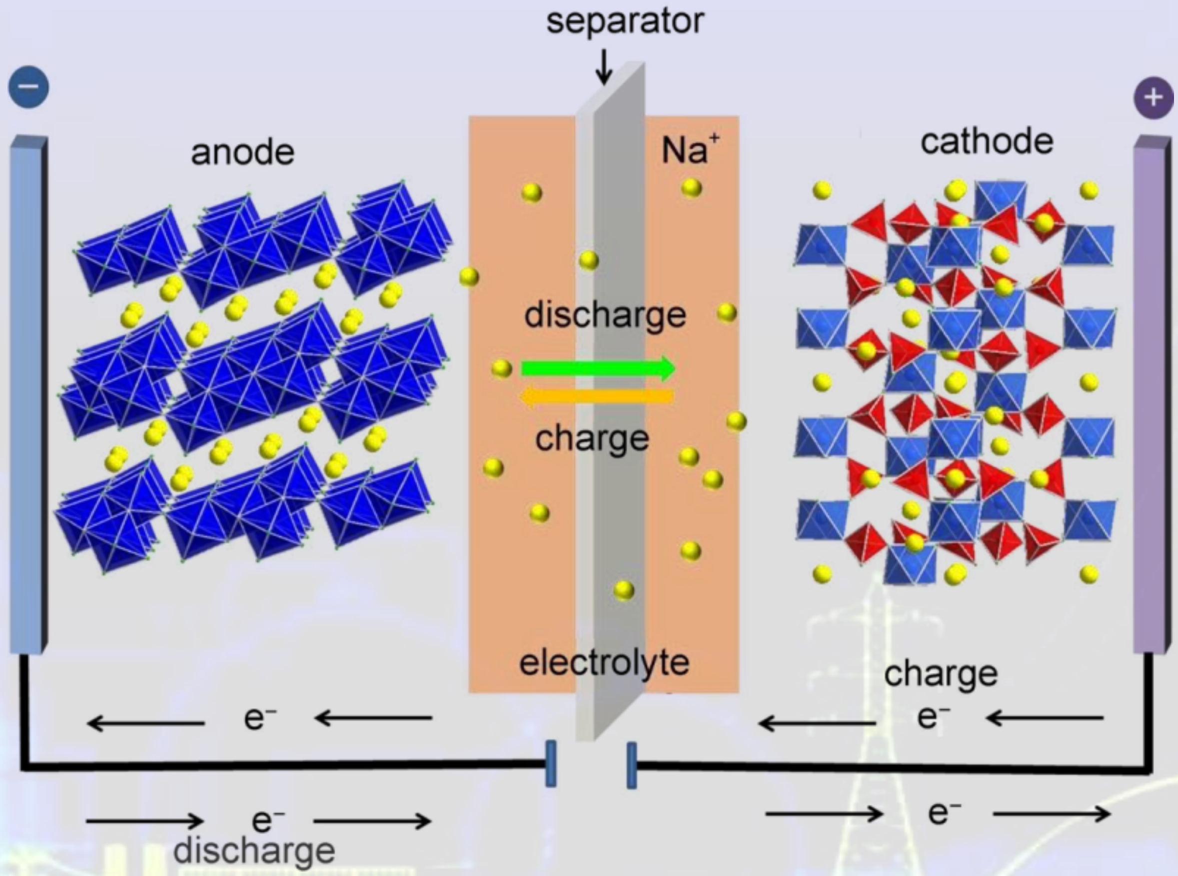
Enegy sources – Electrochemistry
Na-Ion Storage Systems



Schematic illustration of sodium-ion battery working principle
(identical to lithium-ion battery, ‘rocking chair’)

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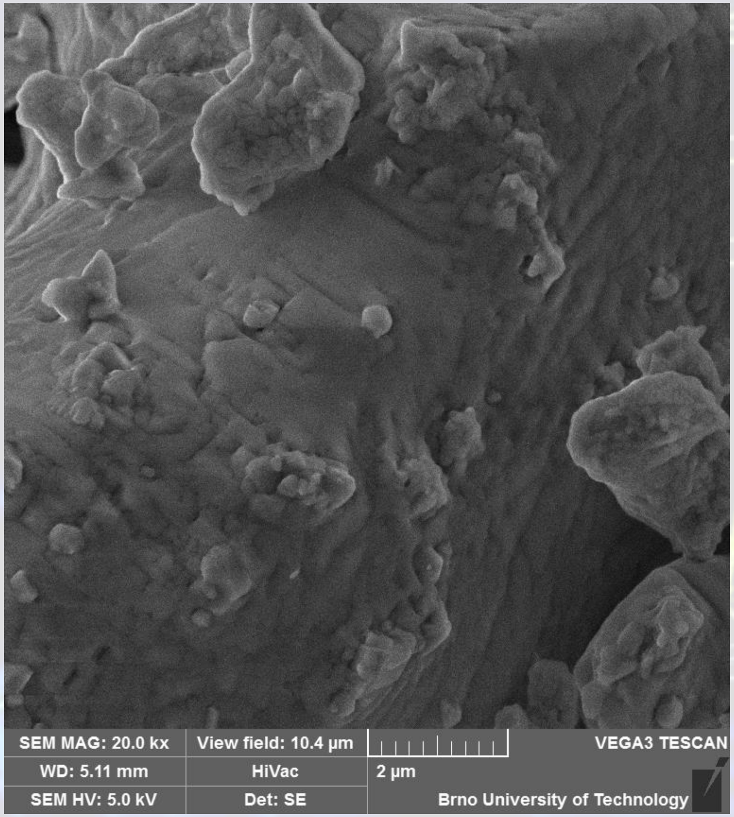
Na-Ion Storage Systems

- Goal is to prepare sodium-titanate material that will be able to accommodate sodium atoms, analogy to comercial available and used (in limited range) lithium titanate oxide (LTO)
- Various ways, solid-liquid or solid synthesis of $Na_xTi_yO_z$ material



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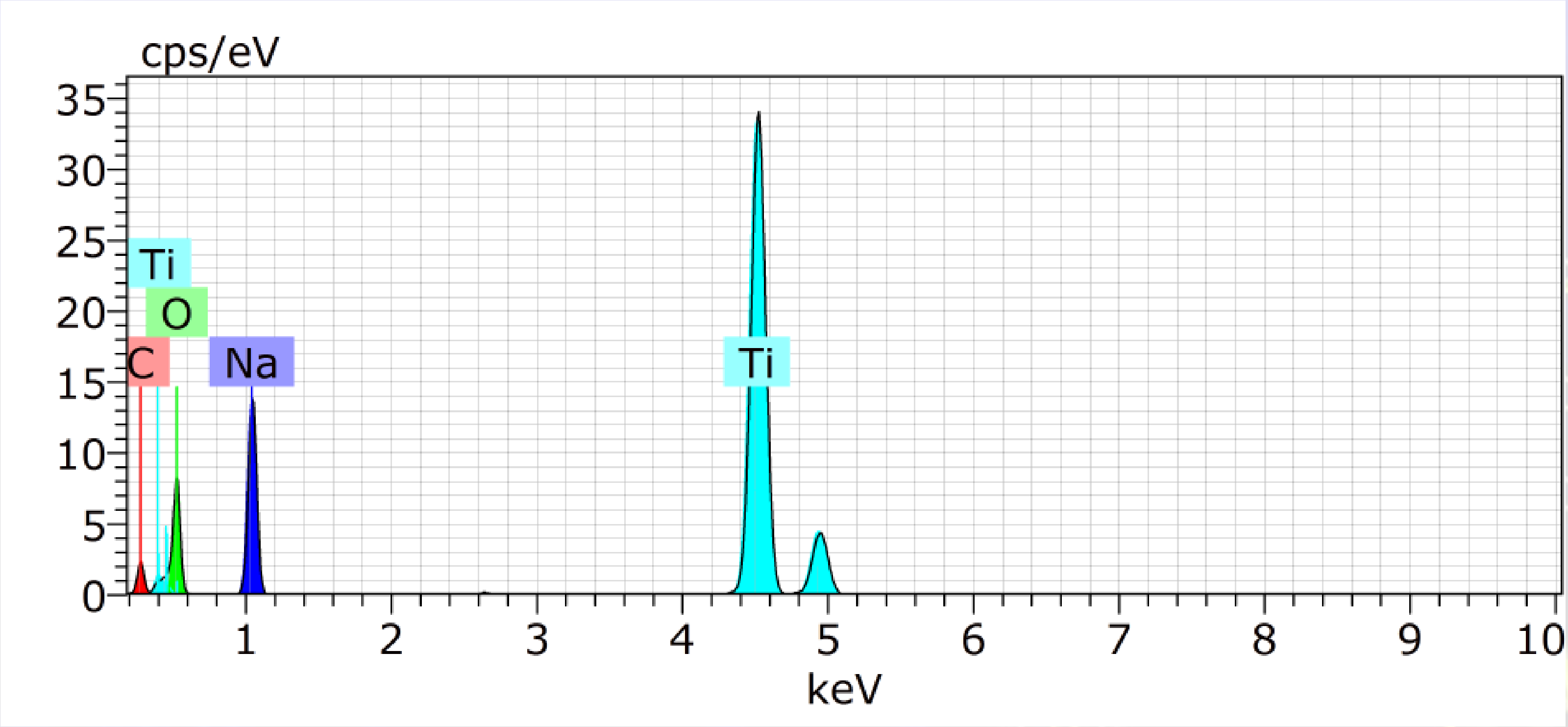
Na-Ion Storage Systems



EDX analytical technique used for the elemental analysis or chemical characterization of a sample

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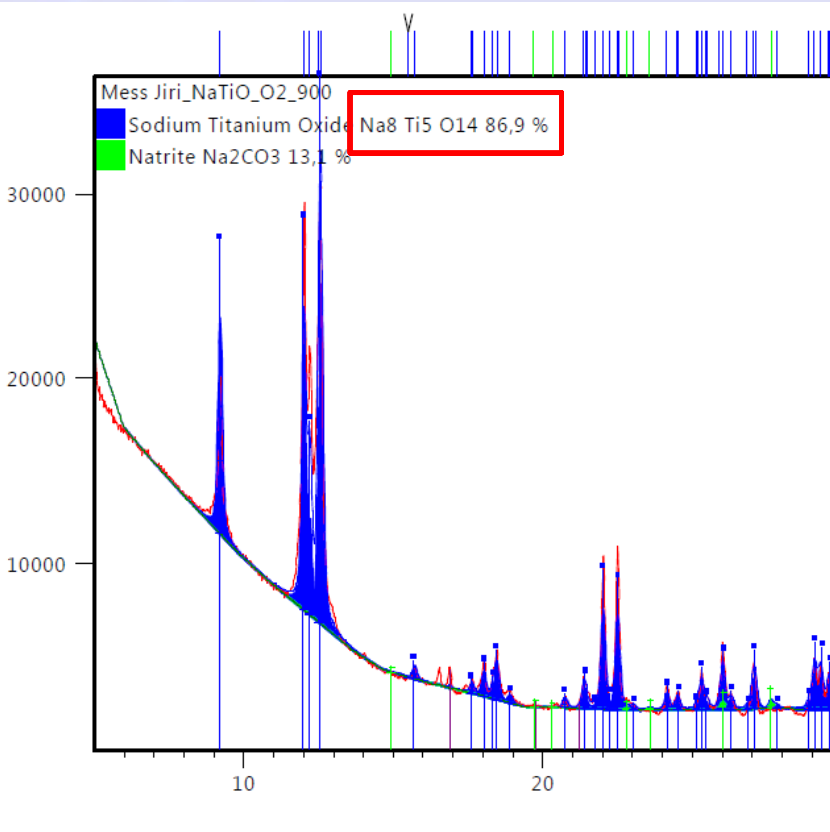
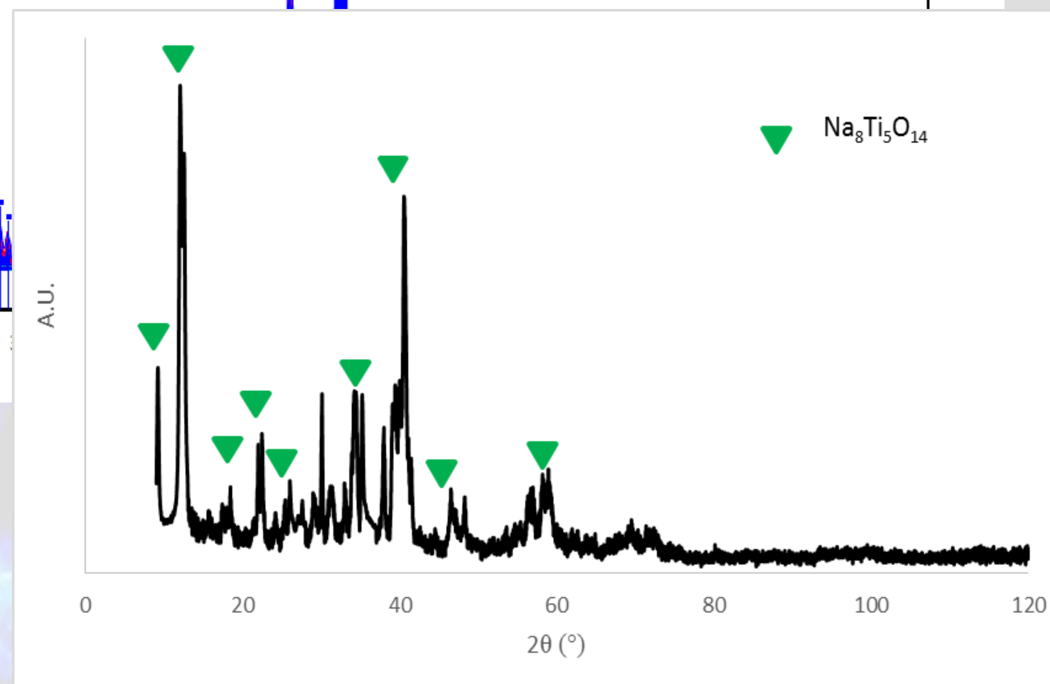
Na-Ion Storage Systems





XRD analysis of NaTiO sample

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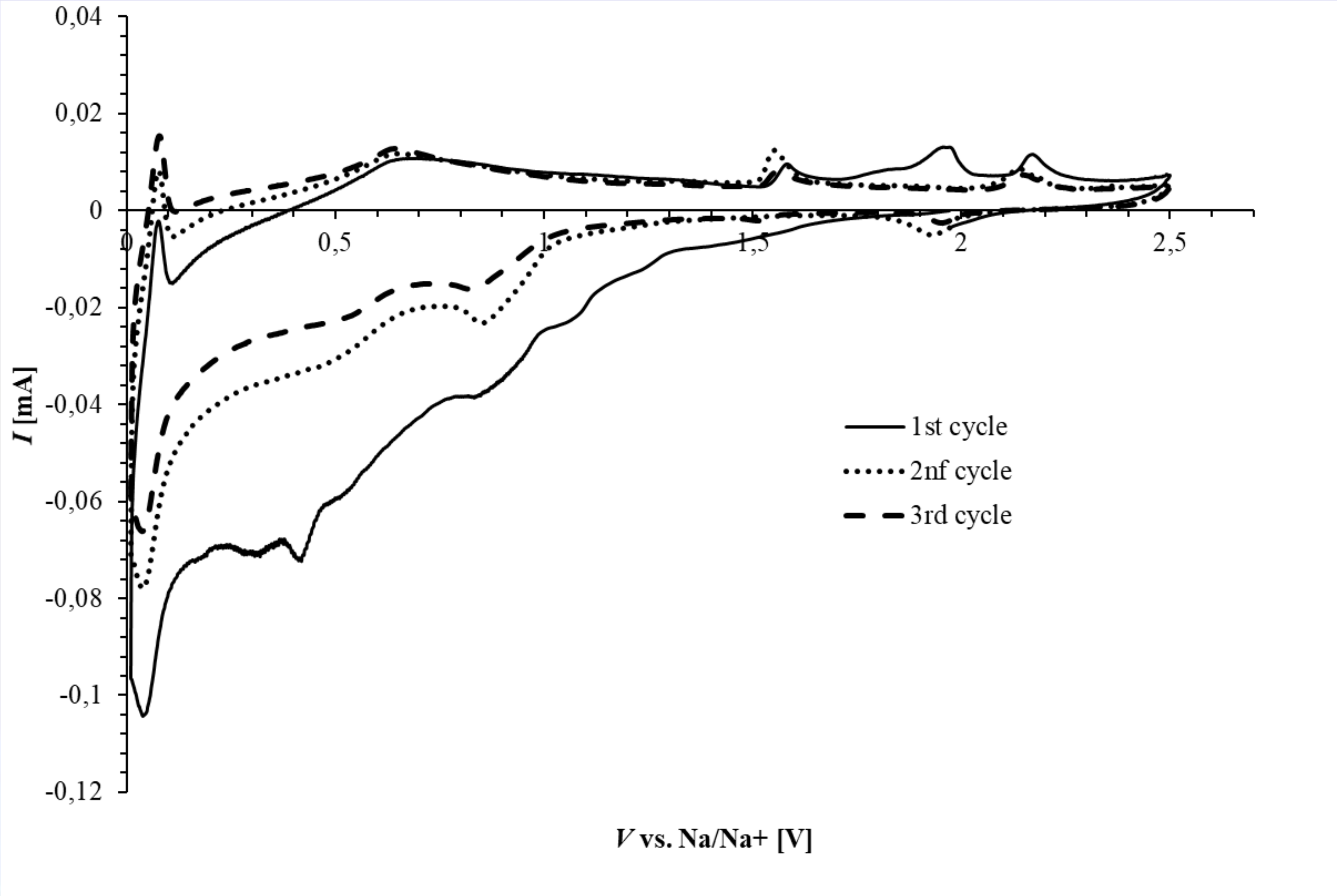
Na-Ion Storage Systems



Cyclic voltammetry (CV) of NaTiO material

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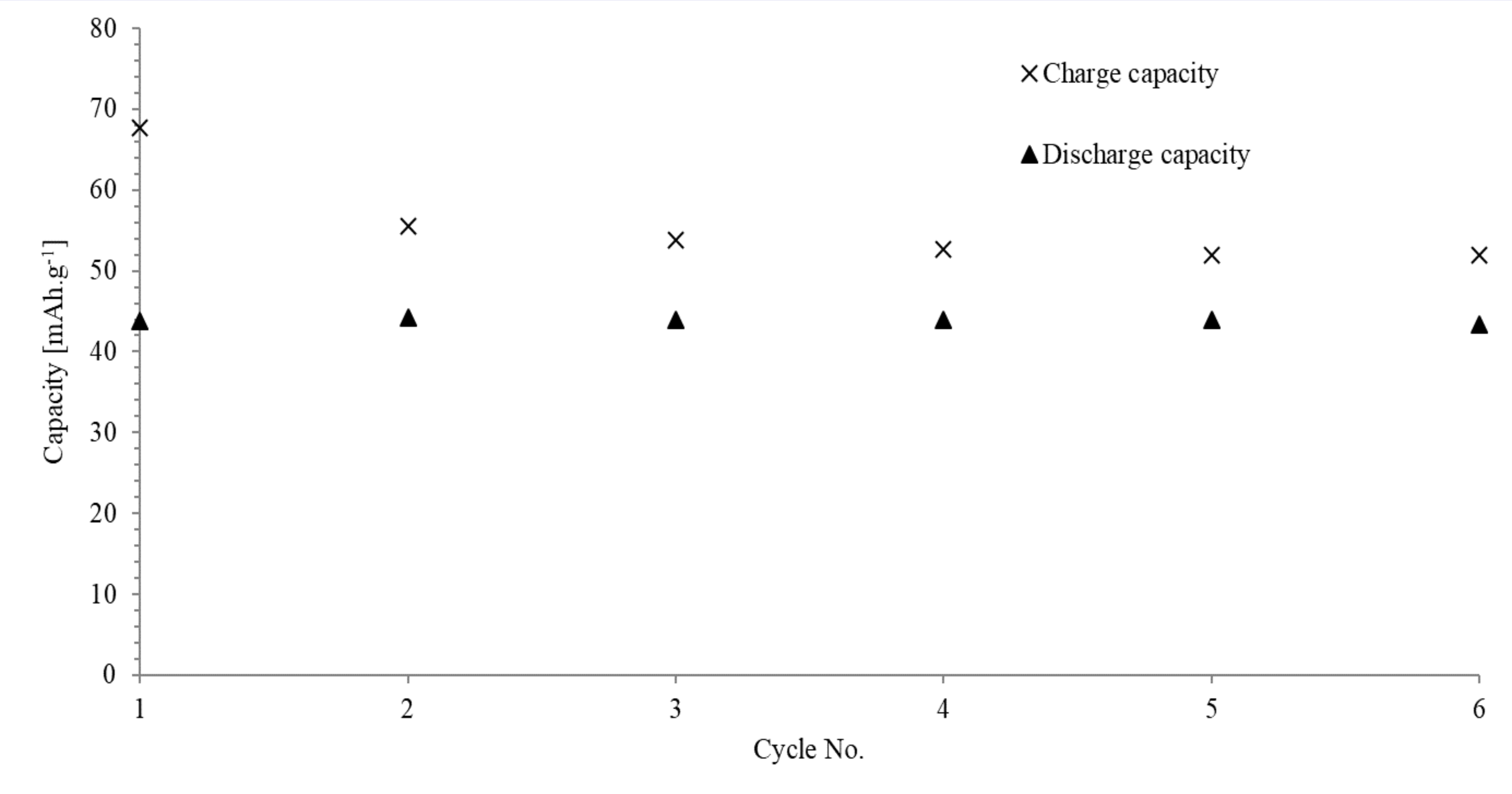
Na-Ion Storage Systems

- Theoretical calculated capacity of $Na_8Ti_5O_{14}$ is ~ 51 mAh/g



First initializating charge-discharge cycles

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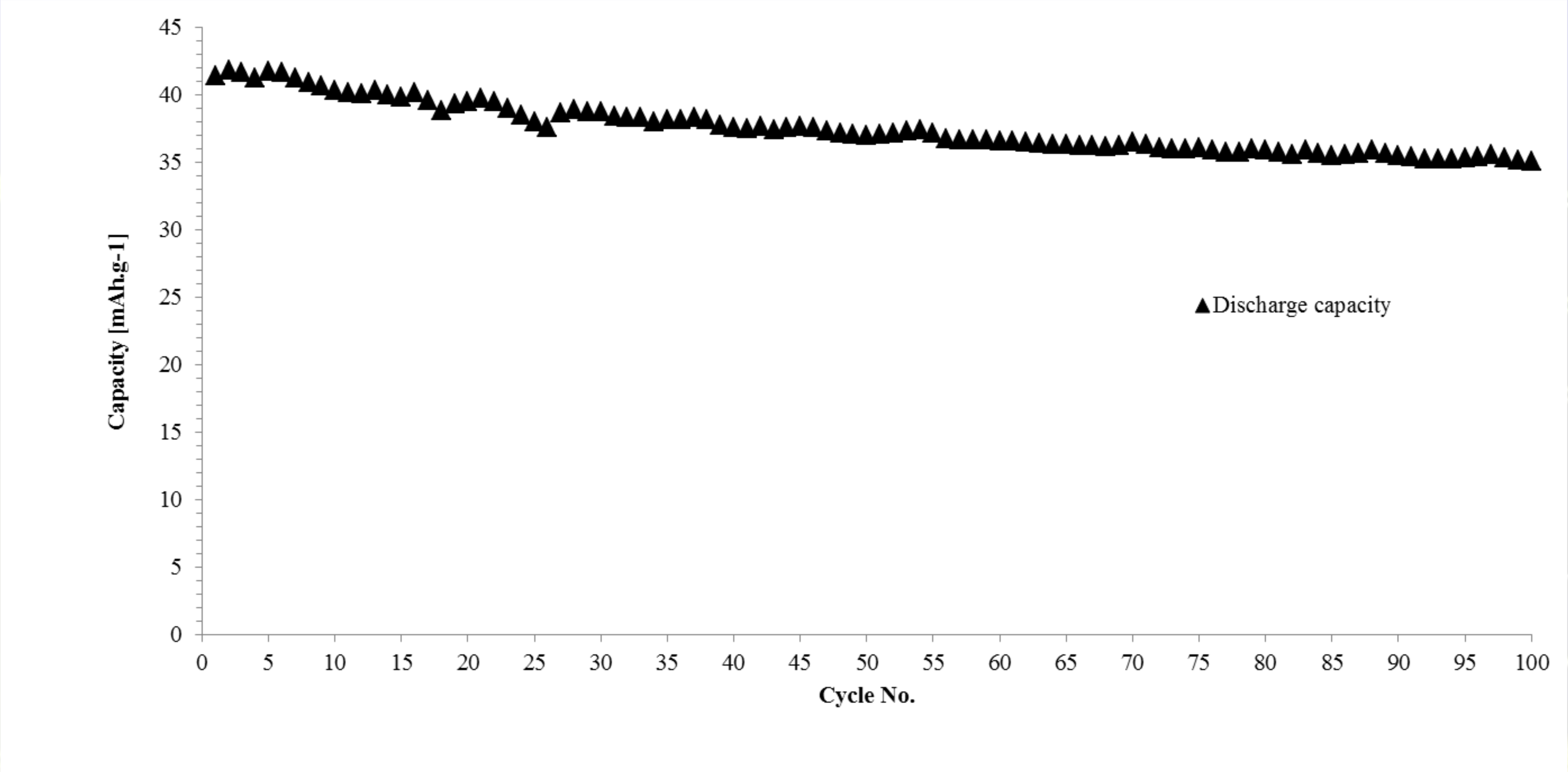
Na-Ion Storage Systems



Galvanostatic cycling at rate 0.2 C

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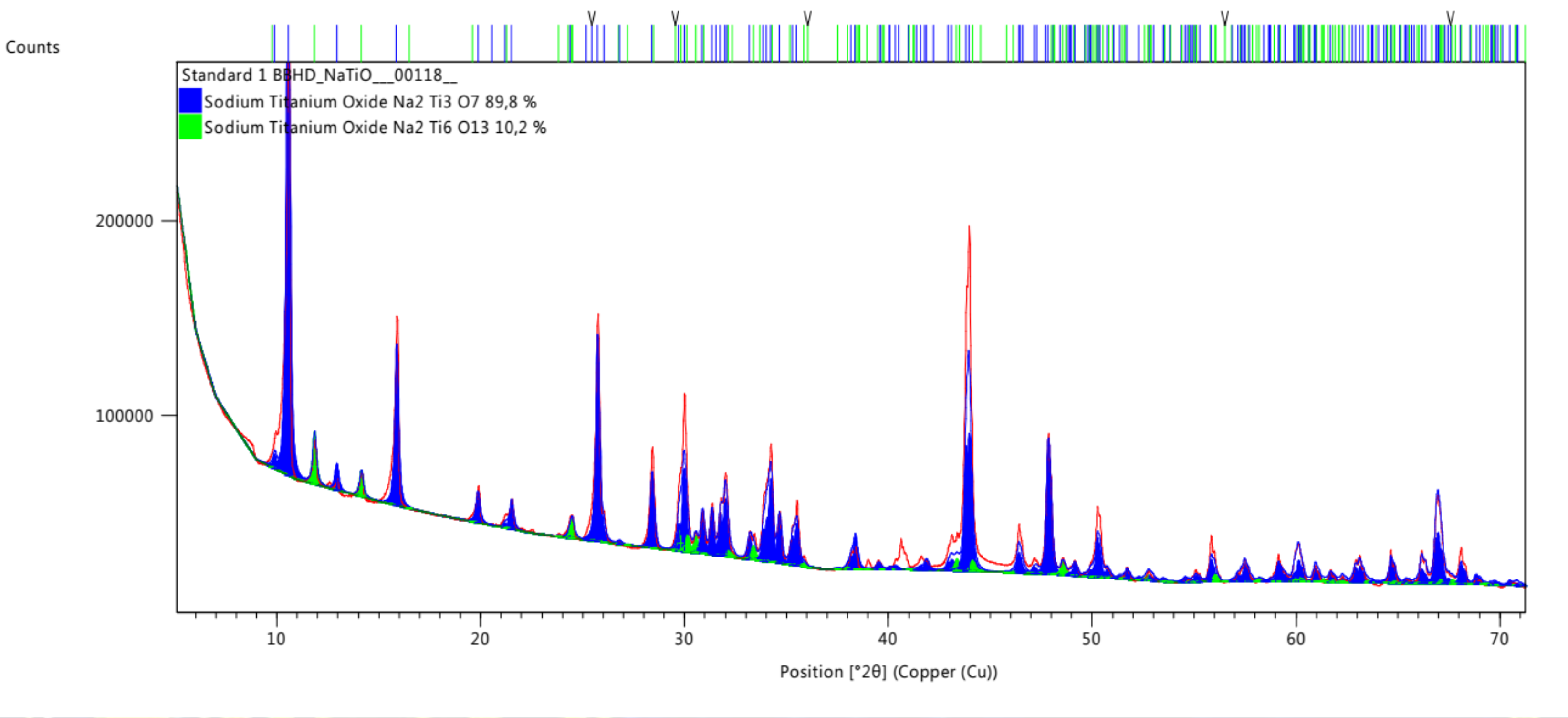
Na-Ion Storage Systems
Current work

- The series of sodium-titanate materials with formula $Na_xTi_yO_z$ were prepared with help of sol-gel process.
- Afterwards the electrochemical testing take a part, from the series of synthesized materials, few exhibited interesting results, as the most promising one with the stoichiometry $Na_2Ti_3O_7$



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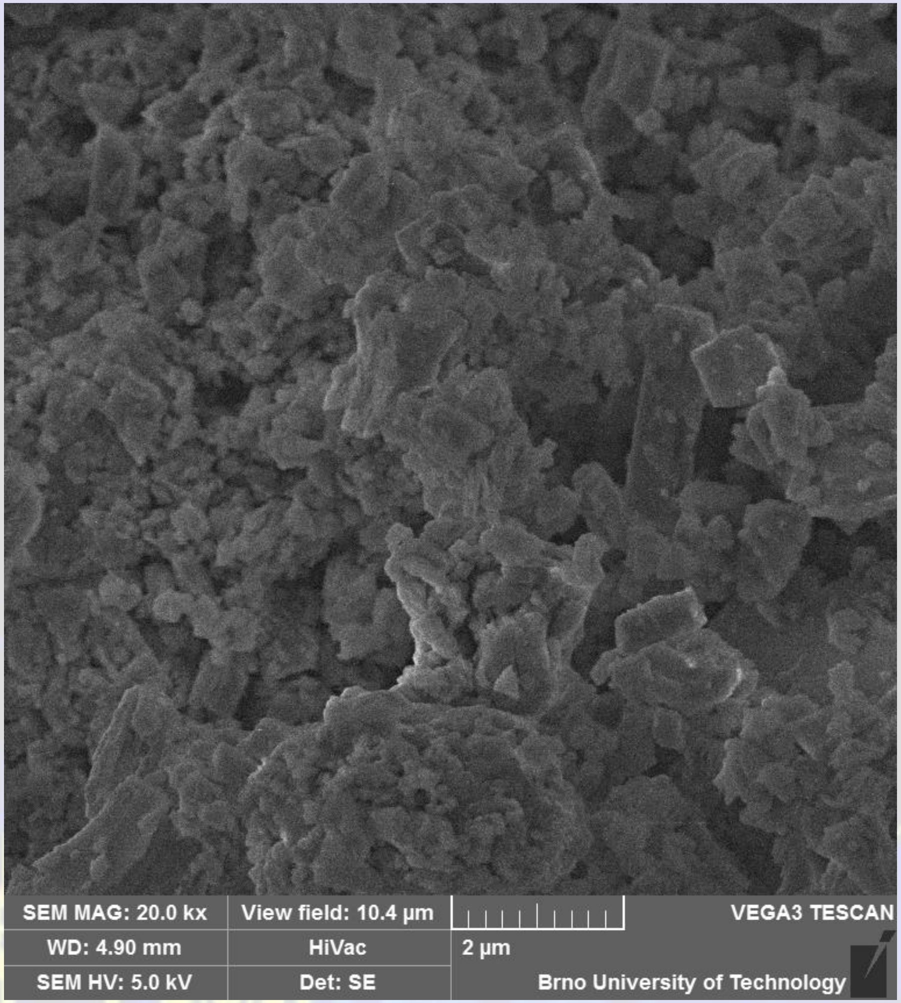
Na-Ion Storage Systems
Current work

- SEM,



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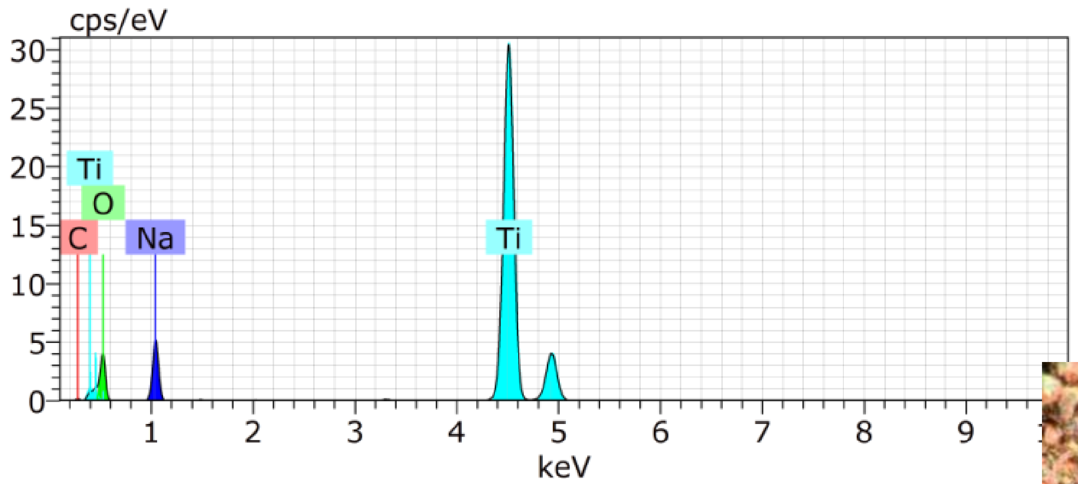
Na-Ion Storage Systems

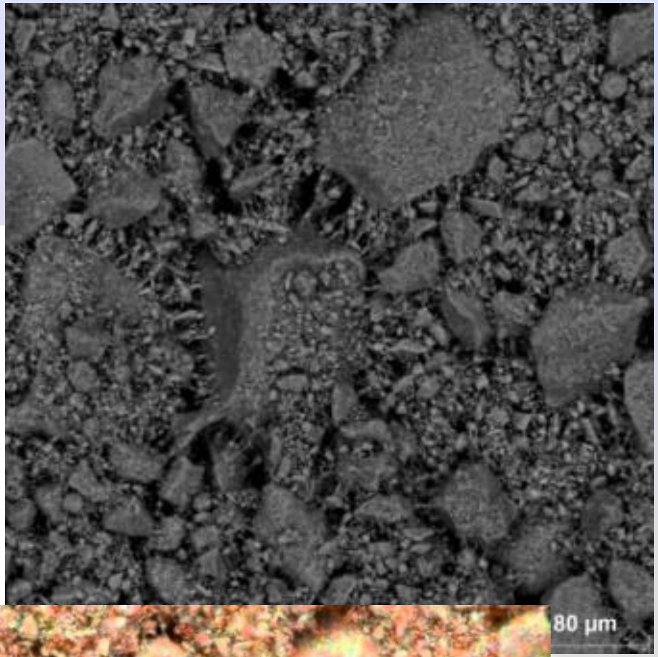
Current work

- EDX



Spectrum: vz 5

Element	AN	Series	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	6	K-series	1,58	3,14	1,13
Oxygen	8	K-series	42,58	63,40	16,41
Sodium	11	K-series	10,51	10,90	2,33
Titanium	22	K-series	45,33	22,56	3,95
		Total:	100,00	100,00	



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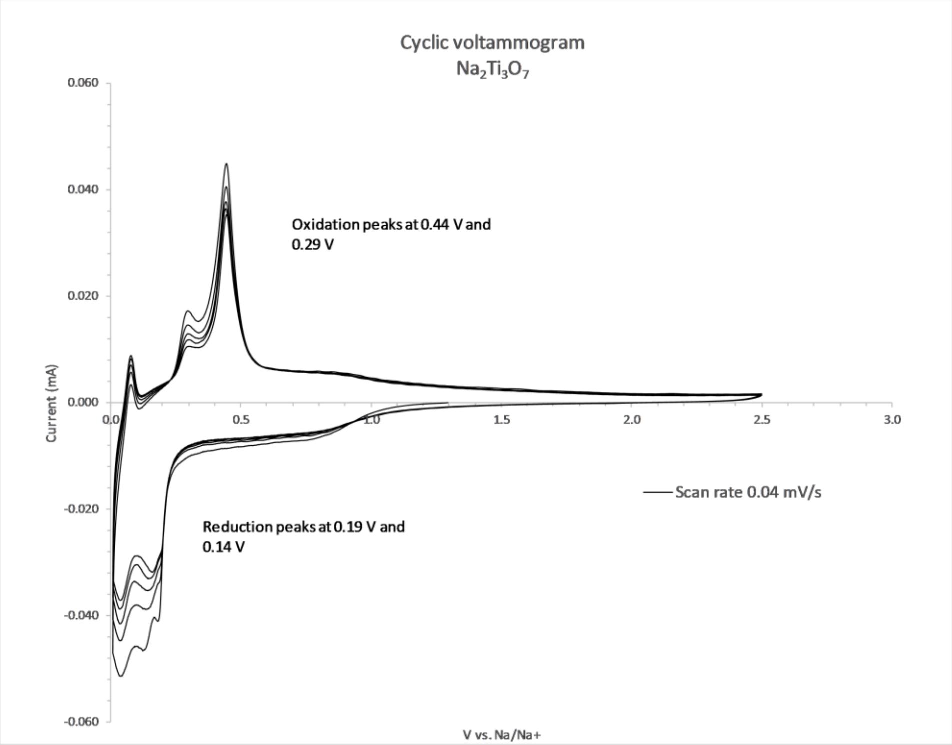
Na-Ion Storage Systems
Current work

- Porozimetry
  - Specific surface area: 2.3 m^2/g
  - Total volume 5.1E-3 cm^3/g
  - Average pore Diameter 1.5 nm
- Results of cyclic voltammetry, another electrochemical testing still do not finished



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Lithium-sulfur accumulator (Li-S)

- ✓ High theoretical capacity 1672 mAh/g
- ✓ Energy density < 3000Wh/kg
- ✓ Highly available
- ✓ Low cost

- × Low potential against Li/Li^+
- × Poor electrical conductivity (insulant)
- × Formation of polysulfides (Li_2S_8 and Li_2S_4 soluble in the electrolyte)
- × Large volume change during cycling

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Lithium-sulfur accumulator (Li-S)

Composition of the basic cell

- Negative electrode:
  - Metallic lithium
- Positive electrode:
  - Sulfur + Amorphous carbon + Binder (PVDF)
- Separator
  - Electrolyte – LiTFSI:$LiNO_3$ DME:DOL

Note:

- **LITFSI** - Lithium bis(trifluoromethanesulfonyl)imide
- **$LiNO_3$** - Lithium nitrate
- **DME** - 1,2-dimethoxyethane
- **DOL** - 1,3-dioxolane
- **PVDF** - Polyvinylidene fluoride

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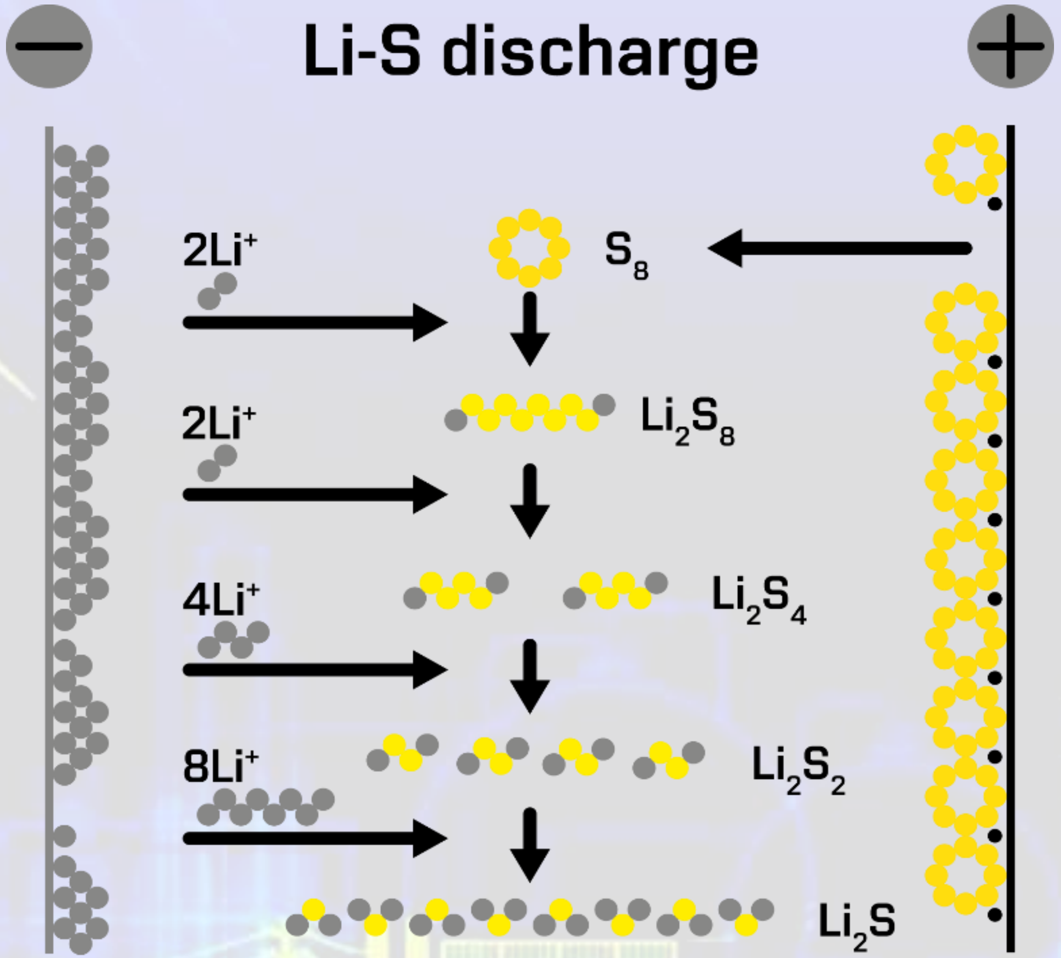
The principle of Li-S cell



Electrochemical reactions occurring in a lithium-sulfur cell during its discharge.

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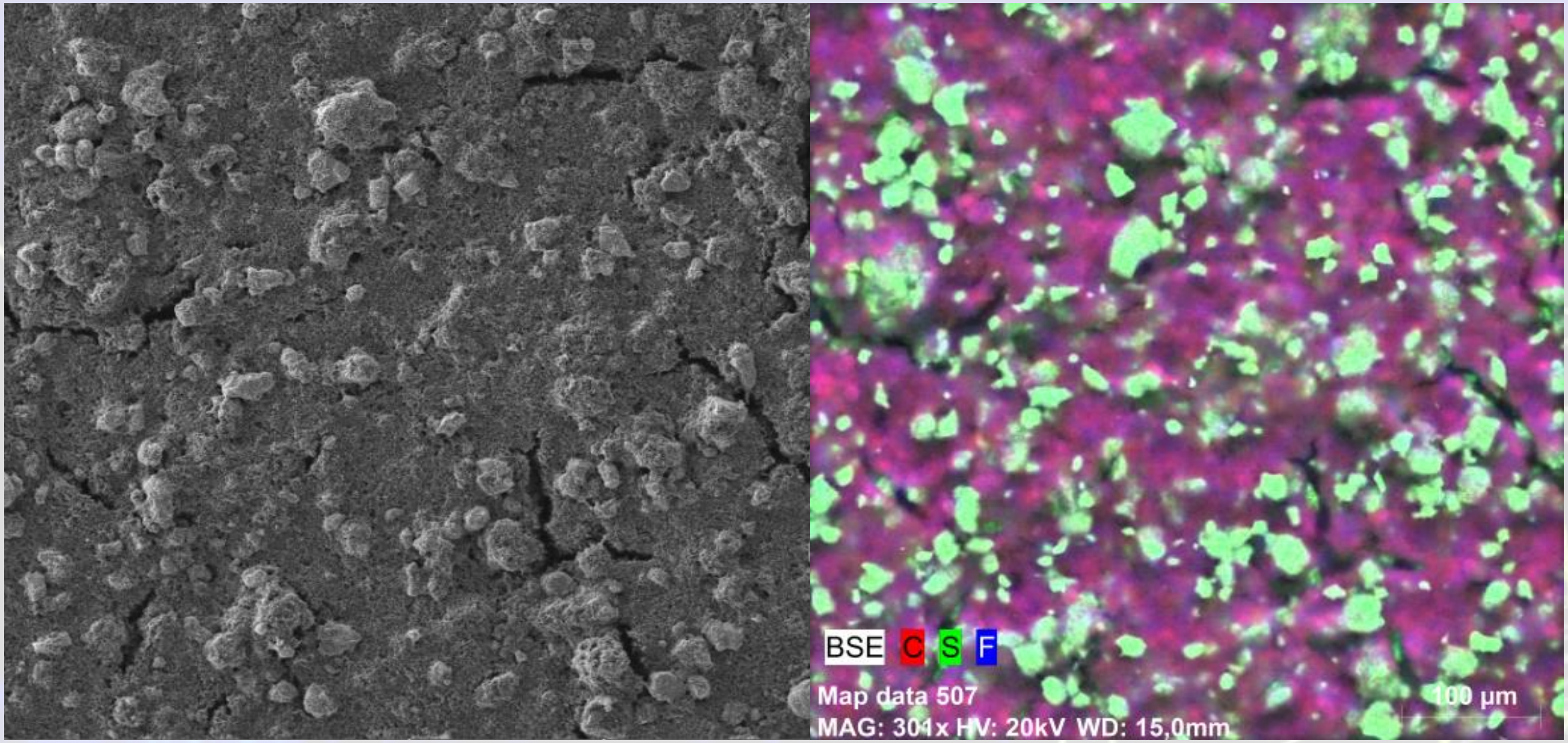
Sulfur electrode under SEM



Sulfur electrode under SEM: A) Surface structure(Mag. 500x, FoV 415 µm) B) Element layout(C,S,F)

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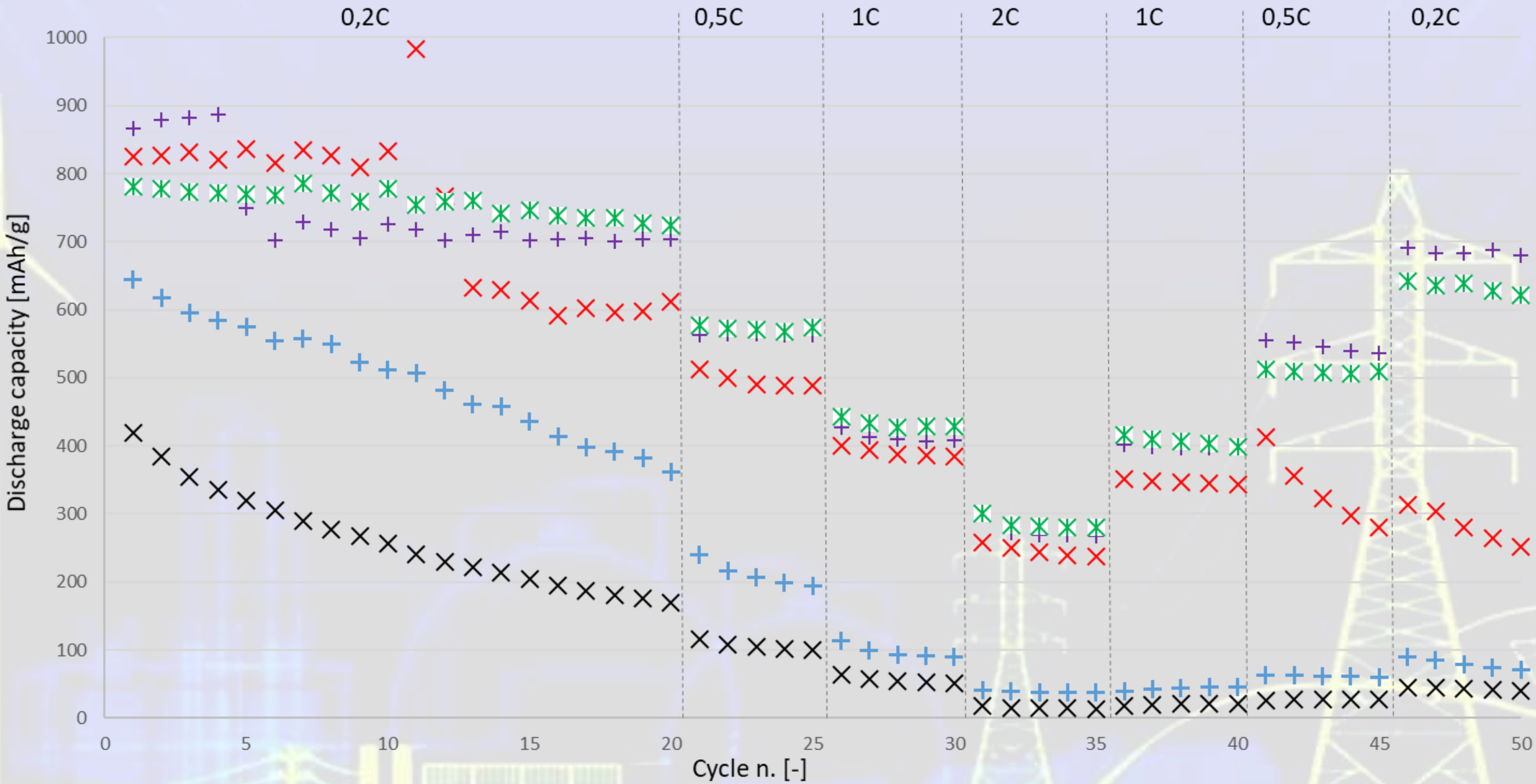
Influence of compression pressure on electrochemical parameters of Li-S cell



Comparison of GCPLs of electrode samples compressed with different pressures (1st measurement)

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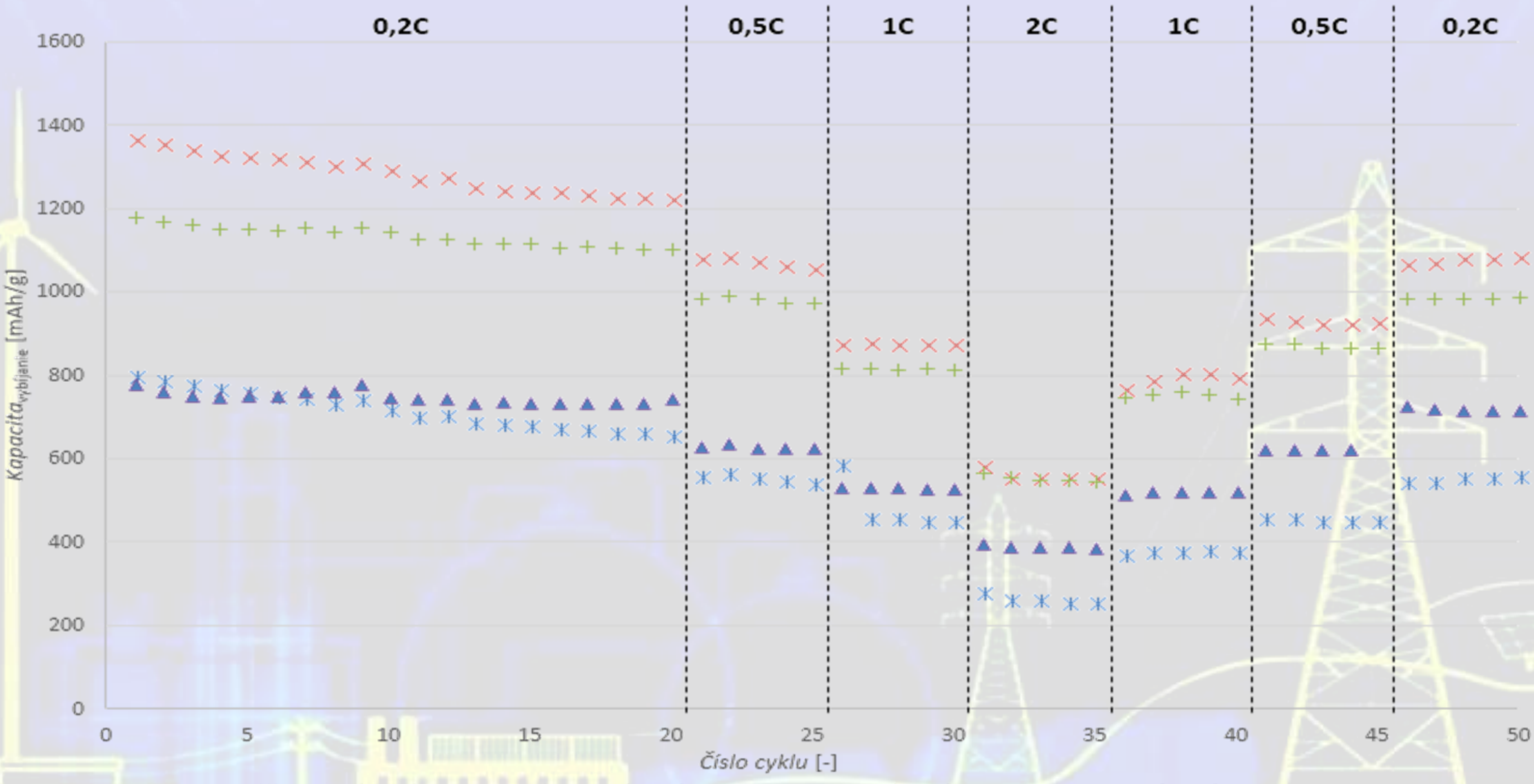

Comparison of GCPLs of electrode samples compressed with different pressures (2nd measurement)

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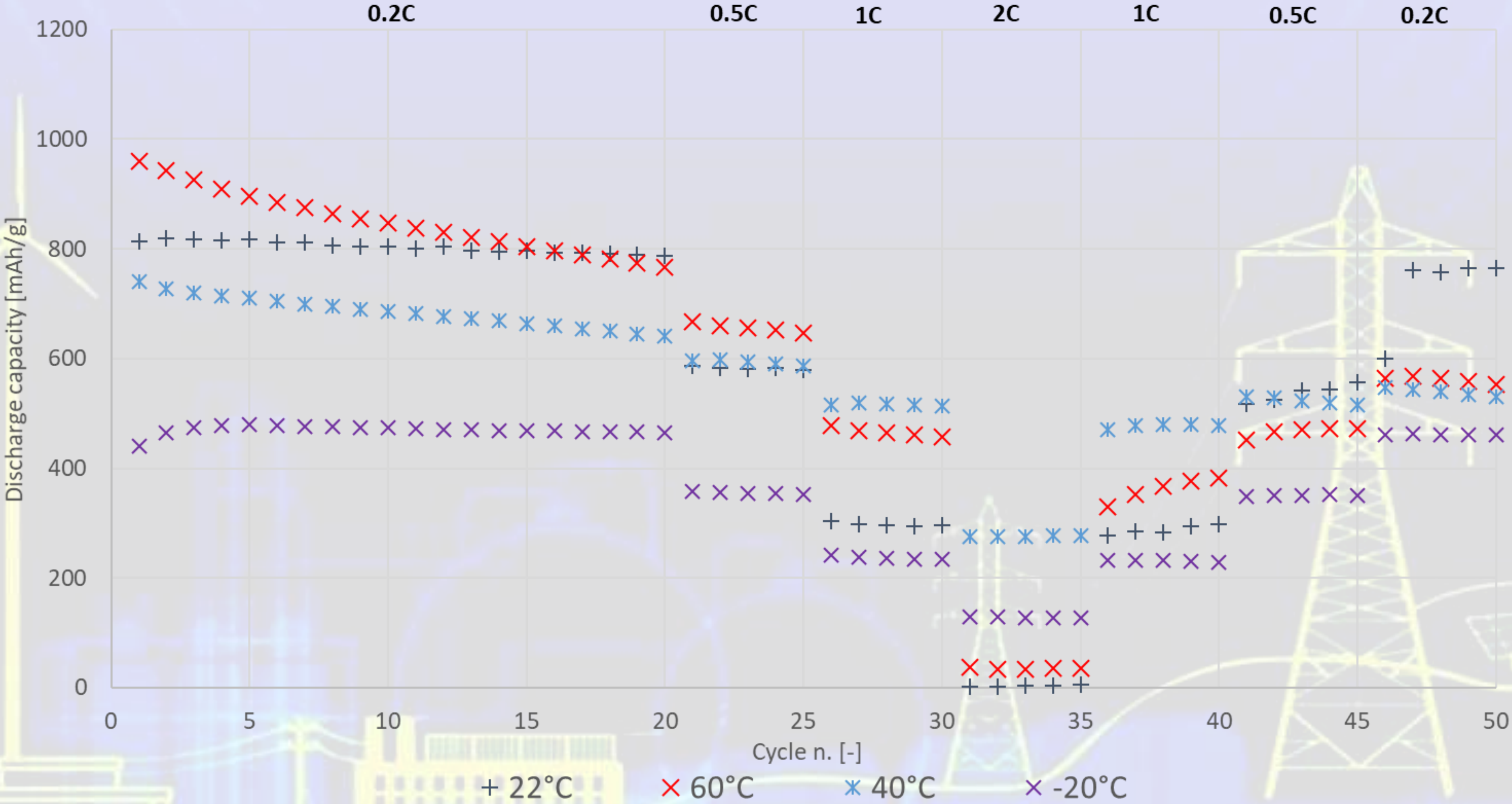
Influence of ambient temperature on electrochemical parameters of Li-S cell



Comparison of GCPLs of electrode samples measured at different ambient temperatures

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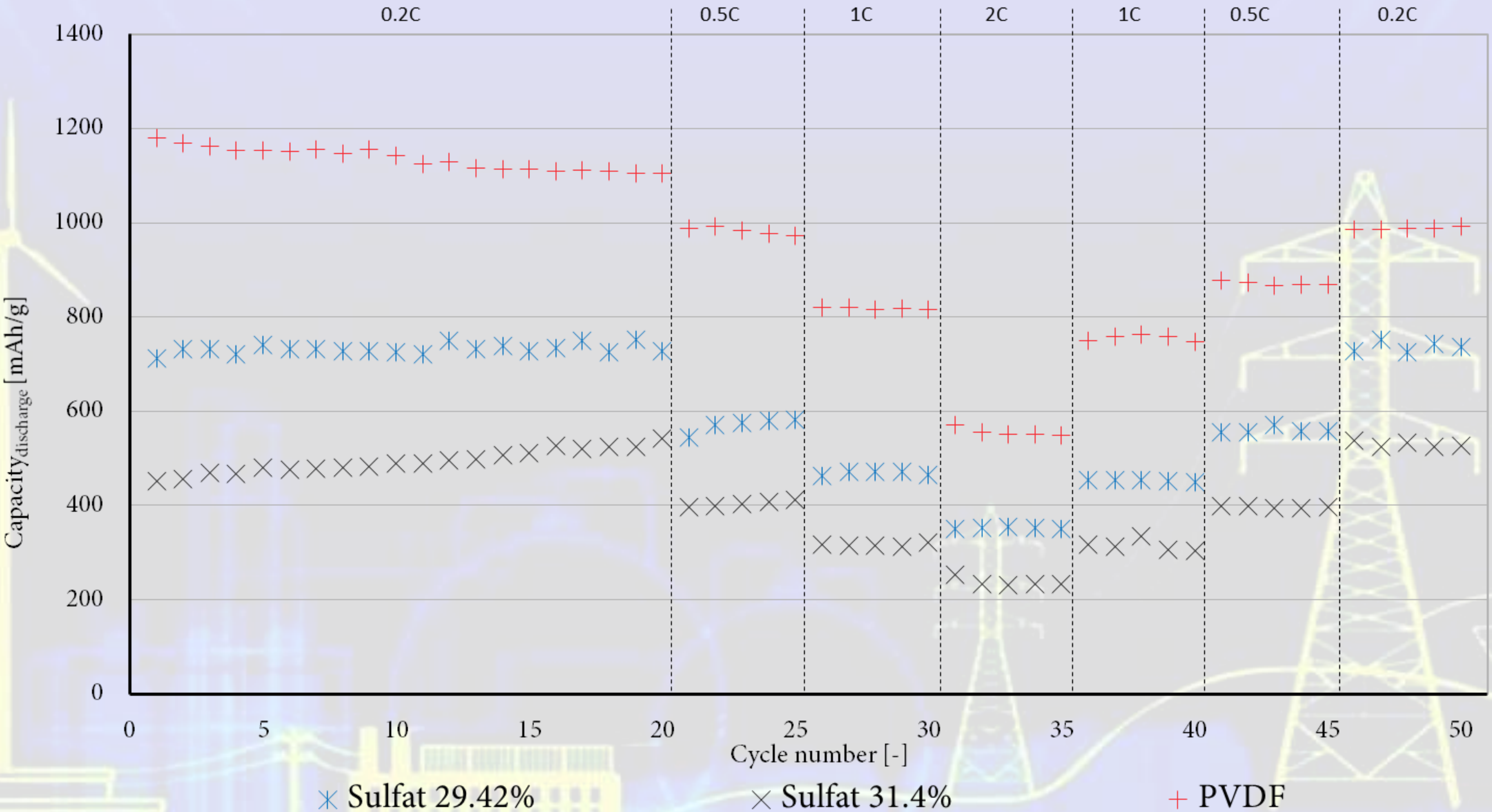
Influence of binders on electrochemical parameters of Li-S cell



Comparison of GCPLs of electrode samples with different binders

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Conclusion

- Was prepared and tested $Na_8Ti_5O_{14}$ anode material, we would like to prepare pure sodium titanate material wit lower stechiometry with molecule formula Na_2TiO_3. This formula reach teoreticla capacity around 188 mAh/g, lower than graphite 372 mAh/g, but for stationary application it is still very interesting solution

- In the year 2017 French start-up company CNRS released prototype of sodium ion rechargeable battery, in cylindrical cell of standard format 18650. The battery reached energy density 90 WH/kg and lifespan over 2000 cycles…

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Brno Univesity of Technology

- Cooperation in EU projects
- Possess the facility background for excellent research
- Offer cooperation within bilateral project regarding stuff and student mobility, Erasmus …
- Help you to provide the invitation letters, conclude memorandum between TUV Wien and BUT, we are widely opened to any collaboration
- You are most welcome to visit us on our international meeting ABAF (Advanced Batteries, Accumulators and Fuel Cells), annually held in Brno, or anytime…

- Jiri Libich, libich@feec.vutbr.cz
- University https://www.vutbr.cz/en/
- Conference meeting https://www.aba-brno.cz/

20th ABAF BRNO 2019
Advanced Batteries, Accumulators and Fuel Cells

FACULTY OF ELECTRICAL ENGINEERING AND COMMUNICATION
department of electrical and electronic technology

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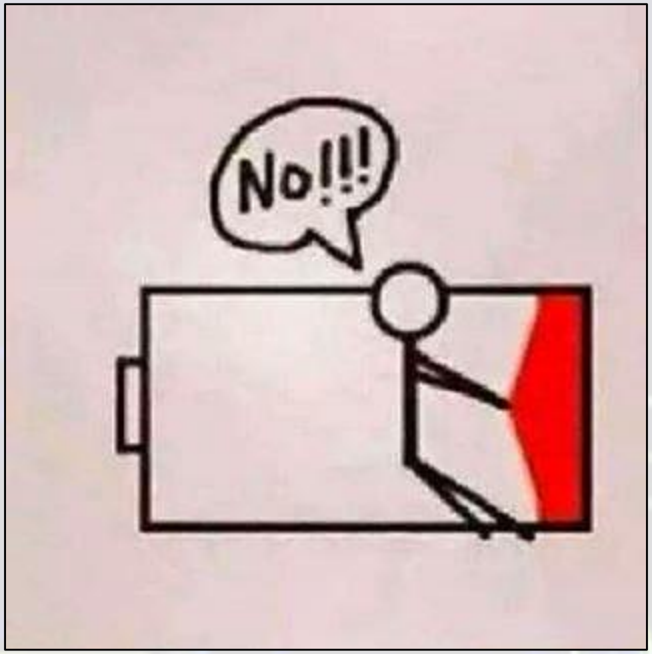
Thank you for your attention!



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