KFM 13: Holistic Structural and Safety Assessment of Lithium-ion and Post-Lithium Cells and their Materials (Experimental Characterisation and Safety Testing)

The focus session is dedicated to the characterization of microstructure, electrochemical, thermal and safety properties of Lithium-ion and Post-Lithium cells and their individual active and passive materials. This is required to obtain quantitative and reliable data, which are necessary to improve the current understanding in order to design and develop better and safer materials and cells. Potential topics include, but are not limited to electrochemical characterization techniques , thermal characterization techniques, safety testing, development of safer materials and cell designs, thermodynamic modelling of materials, modelling of thermal runaway and propagation.

Chair: Philipp Finster (Karlsruhe Institute of Technology)

Time: Wednesday 15:00–16:45 Location: H9

KFM 13.1 Wed 15:00 H9

Are Li-ion batteries safe for 2nd-life applications? - The case of aged cells with SEI growth — •Thomas Waldmann[1,2,4], Gabriela G. Gerosa[1], Jihed Ayari[2,3], Abdelaziz A. Abd-El-Latif[1], Philipp Moosmann[1,5], Max Feinauer[1], Olaf Böse[1], Markus Hölzle[1], and Margret Wohlfahrt-Mehrens[1,2] — [1]Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW), Helmholtzstrasse 8, Ulm, 89081, Germany — [2]Helmholtz Institute Ulm (HIU), Helmholtzstrasse 11, Ulm, 89081, Germany — [3]Karlsruhe Institute of Technology (KIT), Karlsruhe, 76021, Germany — [4]Institute of Surface Chemistry and Catalysis, Ulm University, Albert-Einstein-Allee 47, 89081 Ulm, Germany — [5]Porsche AG, Porschestrasse 911, 71287 Weissach, Germany

Re-using aged Li-ion batteries in 2nd-life applications can increase sustainability. However, there is a lack of knowledge on the safety of aged cells with regard to the underlying aging mechanisms. Room temperature and high temperature aging often lead to growth of the solid electrolyte interphase (SEI) on the surface of graphite or Si/graphite anodes as commonly observed by post-mortem analysis of Li-ion battery cells with physico-chemical analysis methods. We show results on the influence of aging of commercial Li-ion cells in the 1st-life on safety in 2nd-life. Our safety tests (ARC, nail penetration, overcharge, overdischarge) reproducibly show similar or better safety levels for cells with SEI growth in contrast to aged cells with the different mechanism of lithium plating. Possibilities are pointed out to avoid critical mechanisms and for early detection of unsafe behavior.

KFM 13.2 Wed 15:15 H9

Diffraction computed tomography for non-destructive analysis of li-ion batteries — •Vladislav Kochetov — Heinz Maier-Leibnitz-Zentrum (MLZ), Technische Universität München, Lichtenbergstr. 1, 85748 Garching, Germany

This contribution discusses the application of Diffraction Computed Tomography (DCT), including both X-ray and neutron probes, as a powerful method for non-destructive structural analysis in materials science. DCT uses a pencil-beam scanning technique to yield the reconstructed images of internal structure and chemical gradients of materials, extending the traditional imaging approaches. A notable application of DCT is in the study of commercial lithium-ion batteries, where it has been used to resolve inhomogeneities in lithium distribution and structural evolution during cycling. We apply DCT to various commercial battery types, specifically focusing on cylindrical cells with different diameters, featuring diverse chemistries such as NCA, NMC, and graphite anodes. By employing high-resolution DCT, we map lithiation distributions and investigate electrode degradation mechanisms, providing key insights in battery performance and aging. The efficiency of the method, state-of-the-art resolution capabilities, and technique's extension to neutrons are discussed.

[1] V. Kochetov et al, Powder diffraction computed tomography: a combined synchrotron and neutron study, J Phys Condens Matter 33 (10), 2021. [2] D. Petz et al, Lithium distribution and transfer in high-power 18650-type Li-ion cells at multiple length scales, Energy Storage Materials 41, 2021.

KFM 13.3 Wed 15:30 H9

Optical, structural and electrochemical properties of re-synthesized Graphite powder for Anode battery application — •slaheddine Jabri[1], Anna Rollin[2], Sukanya Sukanya[3], René Wilhelm[2], Michael Kurrat[3], Uta Schlickum[1], and Markus Etzkorn[1] — [1]Technische Universität Braunschweig, Institute of Applied Physics, Meldensohn Straße2, 38106 Braunschweig, Germany — [2]Mendelssohnstraße 2 — [3]Technische Universität Clausthal, Institute of Organic Chemistry, Leibnitzstraße 6, 38678 Clausthal-Zellerfeld, Germany

By focusing on preserving the components of Li-Ion battery material through cheaper and envi-ronmental friendly methods, recycling process could introduce scavenged impurities into resyn-thesized material and modify its structural and morphological properties. In this work, we investi-gate the optical, structural and electrochemical properties of recycled Graphite compared to the new material. Our findings reveal that a proper recycling process can remove the Solid Electrolyte Interphase (SEI) layer, which is of significant importance in battery performance. The analysis showed that proper cleaning can significant reduce the amounts of organic and inorganic impuri-ties in the graphite, leading to an improvement in material quality. As a result, the battery perfor-mance can even be enhanced by 89% after 200 charge-discharge cycles compared to the commer-cial base material, demonstrating the potential of recycling methods for improving battery life and efficiency.

KFM 13.4 Wed 15:45 H9

Facilitating Sodium-Ion Diffusion in Fe-Doped Co3O4 for High-Rate Performance — •Yonghuan Fu, Huaping Zhao, and Yong Lei — Fachgebiet Angewandte Nanophysik, Institut für Physik & IMN MacroNano, Technische Universität Ilmenau, 98693 Ilmenau, Germany

Due to its high theoretical capacity, cobalt oxide (Co3O4) has attracted attention to sodium-ion battery (SIB) anodes. However, its low conductivity and poor rate performance have limited its practical application. This work proposes a co-precipitation doping strategy to synthesize iron-doped Co3O4 nanoparticles (FexCo3-xO4 NPs). Both experimental and theoretical results confirm that iron (Fe) doping at octahedral sites within spinel structures is a critical factor in enhancing rate performance. The decreased band gap and enlarged ion transport spacing originate in Fe doping. This effectively facilitates the electron and Na+ transport during discharge/charge processes, delivering an impressive rate capability of 402.9 mA h g-1 at 3 A g-1. The FexCo3-xO4 NPs demonstrate remarkable cycling stability. They maintain a high specific capacity of 786.2 mA h g-1 even after 500 cycles at 0.5 A g-1, with no noticeable capacity fading. This work provides valuable insights into the functional design of high-rate electrodes, offering a promising approach to addressing the critical challenges faced by sodium anodes.

KFM 13.5 Wed 16:00 H9

Reversible Structural Evolution of 3D Vanadium Sulfide Anodes in Sodium-Ion Battery Applications — •Osamah Ali Fayyadh, Yulian Dong, Huaping Zhao, and Yong Lei — Institut für Physik & IMN MacroNano, Technische Universität Ilmenau, 98693 Ilmenau

Sodium-ion batteries (SIBs) are a promising alternative to lithium-ion batteries due to sodium's abundance and wide distribution. However, SIBs face challenges such as low capacity, poor cycling, and sluggish ion diffusion, due to the large ionic radius of Na+. Thus, extensive research has focused on advanced anode materials, among which vanadium sulfides (VSx) have gained significant attention due to their large interlayer spacing, high theoretical capacities, and versatile electrochemical mechanisms. However,VSx suffer from mechanical pulverization, severe volume changes, and limiting their practical application. Here, we demonstrate a 3D micro/nanostructured VSx anode fabri-

cated, achieving a remarkable reversible capacity of 961.4 mAh g-1 after 1500 cycles at 2 A g-1. The sodiation-driven reconfiguration of 3D-VSx into a stable structure mitigates volume changes, enhances ion diffusion, and improves structural stability.[1] The sodiation-driven reconfiguration enhances ion diffusion, mitigates volume changes, and stabilizes the structure. Electrochemical studies and density functional theory calculations reveal significantly improved Na+ storage capabilities, offering a pioneering strategy for developing high-performance SIB anodes with excellent capacity and stability. [1] Y. Dong, Y. Lei et.al. Adv. Energy Mater. 2023, 13, 2204324.

KFM 13.6 Wed 16:15 H9

synergizing nickel (II) oxide-based catalyst for sodium-carbon dioxide battery — •Tzu-Chin Huang, Changfan Xu, Huaping Zhao, and Yong Lei — Fachgebiet Angewandte Nanophysik, Institut für Physik & IMN MacroNano, Technische Universität Ilmenau, 98693 Ilmenau, Germany

Na-CO2 battery is a novel and environmentally friendly green energy device. Conceptually, it demonstrates excellent capabilities. However, during the operation of batteries, undecomposed discharge products, sodium carbonate, accumulate continuously, which is highly insulating, thermodynamically stable, and difficult to decompose. This increases the ohmic resistance within the battery, resulting in high charging potential and excessive polarization, which leads to serious side reactions, such as the decomposition of the electrolyte and cathode material, reducing the battery's reversibility. Herein three Ni oxide-based catalysts, NiO, NiCoO, and CuNiCoO, attached to carbon clothe cathode were synthesized and employed in Na-CO2 batteries. Electrochemical testing demonstrated that CuNiCoO exhibits the best battery stability and long-term performance. This superior performance is driven by the ability of CuNiCoO catalyst to effectively promote the generation and decomposition of discharge products. Cyclic voltammetry (CV) analysis revealed strong redox peaks, underscoring the outstanding catalytic activity of CuNiCoO catalyst. Furthermore, XRD and Raman characterizations confirmed this by showing the appearance and gradual disappearance of sodium carbonate peaks during charge and discharge cycles, indicating remarkable reversibility.

KFM 13.7 Wed 16:30 H9

A Solar Battolyzer Approach: On-Demand Hydrogen Production and Energy Storage in a 2D Niobium-Tungstate Material — Yang Wang[1], •Yu-Te Chan[2], Takayoshi OshimaViola[1], Viola Duppel[1], Sebastian Bette[1], Kathrin Küster[1], Andreas Gouder[1], Christoph Scheurer[2,3], and Bettina Lotsch[1,4] — [1]Max Planck Institute for Solid State Research, Stuttgart — [2]Fritz-Haber-Institut der MPG, Berlin — [3]IEK-9 Forschungszentrum, Jülich — [4]Ludwig-Maximilians-Universität, Munich

In the quest to overcome the limitations of solar intermittency, materials that can simultaneously capture and store solar energy offer promising avenues for clean energy solutions. Here, we introduce the 2D niobium-tungstate $TBA^+NbWO_6^-$ as a novel solution capable of harnessing light energy and storing it either for direct grid integration or as fuel through on-demand hydrogen production. This dual-functionality is central to the emerging concept of "battolyzers," devices that combine battery and electrolyzer capabilities to provide both energy storage and fuel generation. Exposure to light triggers ion intercalation and stable polaron formation within the material, reducing resistance and allowing electron storage for extended durations. Introducing Pt as the catalyst allows the stored electrons to be released to generate hydrogen, demonstrating the material's capability for efficient, on-demand solar energy conversion. Our findings on optoionic processes in $NbWO_6$ lay the groundwork for future solar battolyzers, bridging solar energy storage and hydrogen fuel generation in a single system. [1] Y. Wang et al., J. Am. Chem. Soc. **146**, 25467 (2024)