

MM 15: Battery Materials

Advanced Characterization

Time: Monday 17:30–18:45

Location: H 0106

MM 15.1 Mon 17:30 H 0106

Neutron imaging of Li-ion batteries with fission and thermal neutrons — ●SAMANTHA ZIMNIK^{1,2}, MICHAEL KNAPP^{1,3}, MALGORZATA MAKOWSKA^{2,4}, MARTIN JOHANN MÜHLBAUER^{1,2}, and HELMUT EHRENBURG^{1,3} — ¹Institute for Applied Materials (IAM), Karlsruhe Institute of Technology (KIT), Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany — ²Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Lichtenbergstr. 1, 85748 Garching, Germany — ³Helmholtz Institute Ulm for Electrochemical Energy Storage (HIU), Helmholtzstrasse 11, 89081 Ulm, Germany — ⁴University of Bayreuth, Universitätsstraße 30, 95447 Bayreuth, Germany

Neutron imaging provides outstanding sensitivity to light elements, leading to high contrasts between hydrogen containing materials and metals. Combining imaging with fission and thermal neutrons at a single setup will benefit from the even more selective contrast for hydrogen provided by fission neutrons, while thermal neutrons will serve to reach higher spatial resolution for structure materials surrounding the hydrogen containing materials. Therefore an upgrade of the instrument is currently ongoing to make both neutron energy ranges available at a single setup and benefit from their respective advantages to follow the electrolyte distribution of lithium-ion batteries during operation. The thermal neutron beam option is funded by BMBF in the frame of research project 05K16VK3.

MM 15.2 Mon 17:45 H 0106

Neutrons confirm longer lifetime for Li-ion cells with a be-fitting anode — ●NEELIMA PAUL¹, JOHANNES WANDT², STEFAN SEIDLMEYER¹, SEBASTIAN SCHEBESTA³, MARTIN J. MÜHLBAUER^{1,4,5}, OLEKSANDR DOLOTKO¹, HUBERT A. GASTEIGER², and RALPH GILLES¹ — ¹Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, 85747 Garching — ²Chair of Technical Electrochemistry, Technische Universität München, 85748 Garching — ³VW-VM Forschungsgesellschaft mbH & Co. KG, 73479 Ellwangen — ⁴Institute for Applied Materials, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen — ⁵Helmholtz-Institute Ulm for Electrochemical Energy Storage, P.O. Box, 76021 Karlsruhe

Long battery life is important for stationary storage systems which typically use cells consisting of LFP cathode and a graphite anode similar to needle coke (NC) anode. Such LFP/NC cells typically lose 23% of relative capacity after 4750 cycles and 11% under storage. We report that changing the anode material from NC with a flaky morphology, to a mesocarbon microbeads (MCMB) anode with a spherical morphology can drastically increase the longevity of batteries. In situ neutron diffraction shows that LFP/MCMB cells lose only 8 % of relative capacity under similar cycling conditions and suffer no capacity loss on storage. It also proves that the entire capacity loss is dominated by the loss of cyclable lithium; no other aging mechanisms like structural degradation or deactivation of anode or cathode materials are observed, highlighting the excellent structural stability and superior quality of the investigated cells.

MM 15.3 Mon 18:00 H 0106

Antimatter meets defects: positron annihilation spectroscopy combined with two-component density functional theory to investigate Li₄Ti₅O₁₂ (LTO) — ●C. GROSU^{1,2}, M. SCHUDERER², P. JAKES¹, C. HUGENSCHMIDT³, R.A. EICHEL¹, K. REUTER², and C. SCHEURER² — ¹IEK-9, FZJ Jülich — ²Chair for Theor. Chemistry, TU Munich — ³Heinz Maier-Leibnitz Zentrum, TU Munich

LTO is a well-established anode material with several advantageous electrochemical properties. These depend sensitively on the crystallographic structure and are influenced by the presence of defects within the structure. In fact, defects are purposefully introduced into the structure by tailored synthesis and sintering protocols to increase LTO's electronic conductivity. Little is known to-date about the true

nature of these defects and their exact mechanistic influence. Positron annihilation (PA) is one of the few techniques available to study defect distribution and chemical nature in solid materials experimentally. We perform positron lifetime calculations for bulk spinel LTO using two different self-consistent schemes within the framework of two-component density functional theory. An increased annihilation lifetime on the order of 200 ps is obtained for a positron in a neutral or formally negative defect. In contrast, for positrons in positively charged vacancies we compute lifetimes very close to the 180 ps as for defect-free LTO, likely rendering this type of defect invisible for PA. We discuss these findings in combination with lifetimes and positron coincidence Doppler broadening spectroscopy recently measured at the Garching positron source.

MM 15.4 Mon 18:15 H 0106

Multidimensional operando Analysis of Lithium/Sulfur Cells with X-ray Radiography — ●SEBASTIAN RISSE¹, BENJAMIN KENT¹, ARNE RONNEBURG¹, ANDRÉ HILGER², NIKOLAY KARDJILOV², INGO MANKE², and MATTHIAS BALLAUFF^{1,3} — ¹Institute of Soft Matter and Functional Materials, Helmholtz-Zentrum Berlin, 14109 Berlin — ²Institute of Applied Materials, Helmholtz-Zentrum Berlin, 14109 Berlin — ³Institute of Physics, Humboldt University of Berlin, 10099 Berlin

Lithium/sulfur (Li/S) batteries have a fivefold higher theoretical gravimetric energy density (2680 Wh/kg) than state-of-the-art lithium ion batteries. In addition, the abundant raw materials qualify this electrochemical storage system as one of the most promising candidates for the post-lithium-ion-era. However, the strong fading of electrochemical storage capacity with increasing cycle number is still a major obstacle to a broad technical use despite decades of research. Operando techniques are very suitable tools to gain mechanistic understanding of degradation processes. Especially the simultaneous combination of several independent measurements (multidimensional) while the Li/S cell is in operation allows deep insights into the degradation mechanisms. Here we present results of a novel setup where five different measurements are simultaneously performed. EIS, UV-vis spectroscopy, temperature and X-ray radiography were performed over ten cycles while the cell was galvanostatically charged and discharged. Changes on the macroscopic scale can be correlated to characteristic signals in the EIS and UV-vis spectroscopy as well as the capacity fading curve.

MM 15.5 Mon 18:30 H 0106

Electronic structure of intercalated MXene: towards next generation supercapacitor — ●AMEER AL-TEMIMY^{1,2}, BABAK ANASORI³, SNEHA CHOUDHURY¹, JIAN REN^{1,2}, YURY GOGOTSI³, and TRISTAN PETIT¹ — ¹Methods for Material Development, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Albert-Einstein-Str. 15, 12489 Berlin, Germany — ²Department of Physics, Freie Universität Berlin, Arnimallee 14, 14195 Berlin, Germany — ³Department of Materials Science and Engineering & A.J. Drexel Nanomaterials Institute, Drexel University, Philadelphia, PA 19104, USA

MXenes are a new class of 2D materials consisting of transition metals carbides and nitrides that demonstrated extraordinary features for electrochemical energy storage.

We have investigated MXene (Ti₃C₂) in water using soft X-ray absorption spectroscopy (XAS) at the O K-edge and Ti L-edge before and after urea intercalation. The samples were characterized as dried state in vacuum and in aqueous dispersion solution using a flow cell system.

The intercalation impact of urea molecules between the MXene planes and the effect of aqueous dispersion on their electronic structure will be discussed.

This study demonstrates that XAS performed directly in aqueous media is a powerful method for the characterization of energy-related materials.