MS19-P15 Diffraction and XAFS - the multi-purpose beamline KMC-2

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The beamline KMC-2 at BESSY II, Helmholtz-Zentrum, Berlin, Germany [1], operates a graded SiGe monochromator constructed of two independent crystals. With beam intensity stabilized to an accuracy of 0.3 % and energy resolution of E/ΔE = 4000 in the energy range of 4 – 15 keV, KMC-2 provides high-quality X-ray radiation suitable for demanding applications requiring energy resolution and accuracy. KMC-2 beamline consists of two end stations: "Diffraction" and "XANES". The beamline recently underwent significant modifications, both extending the range of possible applications and allowing for easier handling [2]. Semi-permanently mounted at KMC-2 is the "Diffraction" end station, a six-circle goniometer in Ψ-geometry. The instrument allows a wide range of scattering and diffraction measurements, including grazing incidence diffraction (GID), reciprocal space mapping, diffuse scattering, reflectometry, and powder diffraction. A scintillation point detector with motorized detector apertures allows high resolution experiments. An area detector provides reciprocal space coverage with high counting rates and low background. KMC-2 end station "Diffraction" is frequently used in powder diffraction experiments utilizing its abilities in anomalous scattering or its capability to mount a wide range of dedicated sample environments. KMC-2 "XANES" is a dedicated end station to investigate the short-range environment around selected atomic species in condensed matter by X-ray Absorption Spectroscopy. The end station "XANES" provides capabilities for XAFS, µXAFS, and X-ray fluorescence measurements with a flexible suite of detectors (three ionization chambers, Si-PIN photodiode, energy-dispersive detector) and sample environments with the capability to provide various possibilities for *in-situ* XAFS experiments. As only a short time is needed to switch between "XANES" and "Diffraction", it possible to combine both end stations in the same beam time, making KMC-2 a very versatile beamline especially suited for a wide range of non-standard experiments.

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Keywords: instrumentation, synchrotron, X-ray absorption

MS19-P16 Crystallochemical analysis of ion conductivity in K^+-oxygen containing inorganic compounds.

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A challenge for the materials science is the search for alternative cation-conductive materials, in particular, potassium- or sodium-conductive solid electrolytes, which can be used to create new prospective chemical sources of energy. One of the solutions of this problem is the processing of large amounts of structural data by means of special software based on modern methods of crystallochemical analysis.

The geometry of the migration channels is an important factor which determines the prerequisites for cation conductivity in the structure. In [1, 2] the promising lithium and sodium oxygen-containing compounds, possessing 1D, 2D, or 3D infinite systems of channels (the migration maps) available for Li^+ and Na^+-cations respectively, have been found based on the Voronoi-Dirichlet approach.

In this work, we have analyzed all known ternary and quaternary potassium-oxygen-containing compounds (2729 compounds) from the Inorganic Crystal Structure Database (ICSD version 2015/1). The systems of voids and channels were found in the selected structures using the Voronoi-Dirichlet approach, which is implemented in the ToposPro program package [3]. In total, 231 compounds were found, whose structures allow free migration of potassium ions. Out of the 231 compounds, 53 substances are known as K^+-solid electrolytes, while the remaining 178 compounds, which possess 1D, 2D, or 3D migration maps of K^+ cations (Fig. 1), have not been electrochemically studied so far. They can be used as precursors for the synthesis of new potassium-conductive solid electrolytes.

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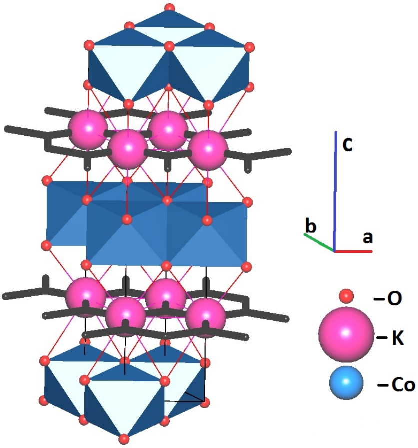

Figure 1. 2D migration map for K^+ cations in $K_{0.61}CoO_2$

Keywords: Crystallochemical analysis, solid electrolyte, Voronoi-Dirichlet approach, systems of voids and channels

MS19-P17 New electrochemical cells for operando neutron diffraction of battery materials

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The interest and frequency of performing operando neutron diffraction experiments for lithium ion batteries has increased significantly over the past few years. A major contributor to this is that the challange to construct an electrochemical cell which balances both electrochemical performance, quality of the obtained diffraction pattern and cost of construction now is addressed. Up until now most work has been performed on, often complex, custom cells built to target a specific feature such as fast cycling at the cost of data quality or data quality with high material loading [1-3].

A significant amount of work has been performed within our group on developing multiple varieties of electrochemical cells for operando neutron diffraction. To this end we have newly designed two vastly different operando cells; a large wound 18650-like cell and a smaller, cheaper coin cell design. The 18650-like wound cell can contain up to 4 g of active material, is able to be cycled at faster rates and provides a diffraction pattern which is of high enough quality to extract accurate structural parameters. It does, however, require expensive deuterated electrolyte and specialised equipment. Alternatively, the coin cell design is cheap, does not require deuterated electrolyte, can provide good quality diffraction and reasonable electrochemical cycling rates. It is anticipated that the coin cell design will make neutron diffraction accessible to more research groups and also presents a viable cell design for operando neutron diffraction studies of sodium ion cells.

Using LiFePO4, LiNi0.5Mn1.5O4 and Li0.18Sr0.66Ti0.5Nb0.5O3 as case study materials this contribution will focus on the operando neutron diffraction results obtained from both cells, thus exploring the core strengths and potential of each design.

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