first of these we present a new divalent Mn succinate framework, $Mn(C_4H_4O_4)$, which undergoes several magnetic phase transitions at low temperature. This compound contains two well separated MnO_6 octahedral sheets with very different connectivity creating two significantly different and well seperated magnetic planes. Neutron powder diffraction has been used to examine the magnetic structure of these planes and this will be presented alongside the important role Mn^{2+} plays in stabilising this unusual structure.

The detailed structures of the transition metal gallates will also be explored. In this fascinating family Fe is found in the trivalent form while all other frameworks contain divalent cations. Despite this, however, initial single crystal structural studies did not indicate any significant differences in the structures of these materials. Here we present a subsequent, more precise, study of these compounds using neutron and synchrotron X-ray diffraction. The differences caused by the presence of di- and trivalent cations and the effect this has on the positions and occupancies of the hydrogens in this structure is described. We also explore the magnetic structure adopted, at low temperatures, by the one dimensional CoO_6 octahedral chains in Co gallate.

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Keywords: neutron, powder diffraction, magnetic structure

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Temperature and pressure evolution of the crystal structure of $A_x(Fe_{1-y}Se)_2$ (A = Cs, Rb, K) studied by synchrotron X-ray diffraction.

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Temperature ($Cs_x(Fe_{1-y}Se)_2$) and pressure evolution of crystal structures ($Cs_x(Fe_{1-y}Se)_2$, $Rb_x(Fe_{1-y}Se)_2$ and $K_x(Fe_{1-y}Se)_2$) was studied using synchrotron powder diffraction. The structure of $Cs_{0.83}(Fe_{0.86}Se)_2$ possesses phase transition on heating related to disorder of iron vacancies in the FeSe layer, as we have shown before [1] and in agreement with other reports [2]. At variance with results reported in [2, 3], we found the transition to be of the first order as evidenced by hysteresis in lattice properties and also agrees with differential scanning calorimentry [1]. At the same time, we have noticed an irreversibility of the temperature dependence of the lattice dimensions and Bragg intensities. We tentatively relate the irreversibility to a mobility of intercalating alkali ions; such a mobility may seriously affect apparent temperature evolution of the crystal structure.

Diffraction experiments as a function of pressure allowed to recover equation of states and, for the first time, provide with experimental estimates of the bulk moduli. We have also seen that the Bragg reflections indicative of vacancies ordering in the FeSe layer do not disappear up to ~120 kbar. Albeit refinement of crystal structure suffers from limited quality of diffraction data collected in diamond anvil cells, no clear anomaly for $Cs_{0.83}(Fe_{1-y}Se)_2$, $Rb_{0.85}(Fe_{1-y}Se)_2$ and $K_{0.8}(Fe_{1-y}Se)_2$ ($y \sim 0.14$) was observed around 80 kbar, where transport experiments indicate an offset of superconductivity. Albeit definitive answer can only be obtained from diffraction experiment at high pressure and very low temperatures, our data indicates that ordering of vacancies in the FeSe layer may be present in both superconducting and non superconducting states.

Keywords: superconductor, synchrotron X-ray diffraction, Pressure

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Neutron diffraction on functional materials using special sample environments

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The high-resolution neutron powder diffractometer SPODI (FRM II / Garching n. Munich) offers a range of possibilities for in-situ materials characterisation under special sample environmental conditions.

Bulk samples of ferroelectric ceramics were investigated under the influence of high electric fields up to 7 kV/mm (35 kV) in a SF6 gas atmosphere to establish correlations between the macroscopic poling behaviour and corresponding structural changes. Here we report investigations on lanthanum doped lead zirconate titanate (PLZT) with compositions around the morphotropic phase boundary [1] and also on a bismuth sodium titanate based system (BNT-BT-KNN) [2]. The neutron diffraction studies are accompanied by X-ray and synchrotron diffraction as well as transmission electron microscopy. In the system BNT-BT-KNN at composition 92-6-2, the field induced macroscopic strain can be explained by a phase transformation during the poling process. At 6 kV/mm the transition to a rhombohedral phase was identified by corresponding superlattice reflections, arising from a superstructure in the tilting angles of the oxygen octahedra around Ti/Zr atoms. The data were analysed by Rietveld refinement. In lanthanum doped PZT samples with different Ti/Zr compositions, systematic changes in the response to the electric field were observed for different compositions across the morphotropic phase boundary: different changes of lattice distortions and anisotropic displacement parameters of the lead cations were found.

Other in-situ neutron diffraction studies on SPODI comprise e.g. the structural changes in lithium-ion batteries during charging/discharging which were investigated in-operando by a potentiostat or deuterium charging of hydrogen storage materials with D_2 pressures up to 100 bar in cooperation with the Joint Research Centre of the European Commission in Petten [3]. The anisotropy of material reactions under mechanical load is studied with a rotatable load frame, allowing the selection of any direction of the applied field with respect to the diffraction plane.

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