

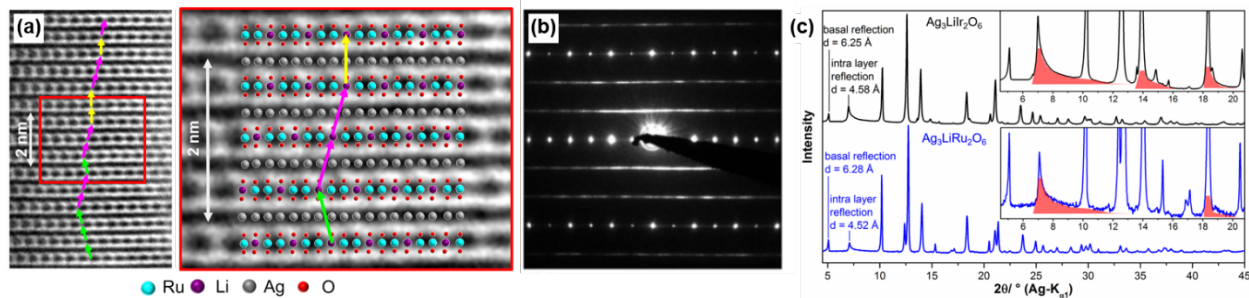
# Crystal structure solution and refinement of stacking faulted inorganic materials using the TOPAS software

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Many crystal structures contain two-dimensional layered structural motifs (atoms, ions or molecules) periodically repeated in the third dimension, giving the crystal a distinct stacking sequence. In real crystals (including technologically important materials like battery electrodes and alloys), the stacking order can deviate from ideal periodicity. The stacking sequence becomes non-uniform (Fig. 1 a, green, magenta and yellow arrows), and a microstructure is created. Transitions between different stacking sequences appear as *stacking faults* and can have a huge impact on the diffraction pattern. These defects lead to diffuse elastic scattering generated at arbitrary positions in reciprocal space, and consequently, scattered intensity is no longer observed only at distinct reciprocal lattice points. In three-dimensional reciprocal space, new features such as streaks (Fig. 1 b) or superstructure reflections appear. Often, the type of faulting can be different for individual single crystals of the same batch of a certain material. By simultaneously collecting scattering patterns averaged over a large number of crystallites, as with X-ray powder diffraction (XRPD) or neutron powder diffraction (NPD), structural details can be obtained that are very representative of the bulk properties. Since powder diffraction is the one-dimensional projection of three-dimensional reciprocal space, the above mentioned effects can appear as new peaks, peak shifts, peak asymmetries (Fig 1 c, red highlighted reflections), and/or peak broadening all over the powder pattern. Improvements in computational hardware and software algorithms<sup>1,2</sup> enable quantitative analyses on the degree of faulting by recursive routines. In this talk, modelling and quantification of stacking fault disorder in polycrystalline, inorganic materials using the TOPAS<sup>3</sup> software is shown. Refinement strategies for stacking faulted quantum spin liquids:  $\text{H}_3\text{LiIr}_2\text{O}_6$ <sup>4</sup>,  $\text{Ag}_3\text{Li}(\text{Ir}/\text{Ru})_2\text{O}_6$ <sup>5</sup>; super ion conductors:  $\text{Li}_3\text{HoBr}_{6-x}\text{I}_x$ <sup>6</sup>, the potential ferroelectric material  $\text{SnTiO}_3$ <sup>7</sup>, NCA<sup>8</sup> (nickel, cobalt, aluminum hydroxide) and NCM (nickel, cobalt, manganese hydroxide) type battery material precursors; thermoelectric  $\text{Cu}_{0.50}\text{Sn}_{0.25}\text{Zn}_{0.25}\text{S}$ <sup>9</sup> and for spin coated  $\text{H}_3\text{Sb}_3\text{P}_3\text{O}_{14}$ <sup>10</sup> thin films will be presented.



**Fig. 1.** (a) HRTEM micrographs of  $\text{Ag}_3\text{LiRu}_2\text{O}_6$  along zone axis  $[110]$  and overlay of a supercell of  $\text{Ag}_3\text{LiRu}_2\text{O}_6$  with an excerpt of HRTEM micrograph, (b) PED pattern of  $\text{Ag}_3\text{LiRu}_2\text{O}_6$ , (c) Comparison of the measured XRPD patterns of  $\text{Ag}_3\text{LiIr}_2\text{O}_6$  (black pattern) and  $\text{Ag}_3\text{LiRu}_2\text{O}_6$  (blue pattern). Characteristic triangular peak shapes indicating structural disorder are highlighted by red background color.

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