B1: Investigation of phase formation and phase constitution in the systems Li-Mg-Al-O and Li-Al-Mn-O with special focus on spinel solid solutions

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Project area B1 of the DFG project SPP 2315 aims to modify the slag system of current pyrometallurgical recycling processes of Li-ion batteries to enable the recovery of Li. $LiAlO_2$ is a promising engineered artificial mineral (EnAM) to be separated from the rest of the slag to recover Li. To make this approach feasible, the formation of $LiAlO_2$ must be maximized and the formation of other Li-containing phases must be supressed. Therefore, it is essential to understand the phase formation in the slag system. A special focus is on the formation of spinel solid solutions, which are known to be a concurring process to the formation of $LiAlO_2$ during the primary crystallization.

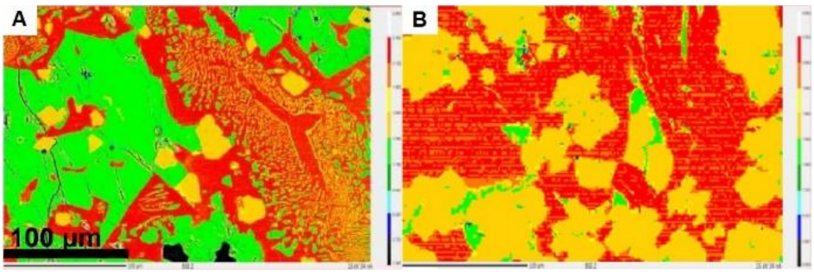

Figure 1: Two different domains in a synthetic slag system. A: Dominant $LiAlO_2$ (green) and suppressed spinel (yellow). B: vice versa. Red: Ca-Al-silicate matrix.

The two quaternary subsystems Li-Mg-Al-O and Li-Al-Mn-O, which are part of the complex oxide slag system (Li, Mg, Al, Si, Ca, Mn)-O, are the most relevant to understand the $LiALO_2$ / spinel (ss) formation. The investigation of these subsystems will be conducted using combinatorial deposition of thin film materials libraries, accompanied by compositional and crystallographic high-throughput characterization as well as mineralogical investigation and characterization. In the system with Mn a special focus will be on the influence of the speciation (MnII – MnIV) on the phase reactions. Annealing of the materials libraries will be carried out at relevant temperatures to gain information about the phase formation and kinetic behavior of the systems. The result will be existence diagrams of the two multinary oxide systems. This approach aims on specifying “regions of interest” for solidification experiments. The compounds and the morphology of selected solidified melts in the system Li, Mg, Al, Mn, O will be analyzed with PXRD and EPMA. The Li contents of the samples will be determined by means of XPS, SIMS, and for selected samples atom probe tomography. These results in combination with the material libraries will be used for mineralogical characterization including a comparison of the reactions on the micro-scale (thin film material library) and the macro-scale (melt experiments) with pico-liter printing experiments as intermediate step on demand to gather additional information on the kinetics of these systems.