Spatially resolved analysis of garnet-type Li-ion conductors using LIBS: Investigation of the H-content

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Since current Li-ion batteries suffer from problems caused by the chemical instability of their utilized organic electrolytes, it is of major interest to replace them by more stable inorganic solid ion conductors. Due to their high Li-ion conductivity and superior stability properties, cubic $Li_7La_3Zr_2O_{12}$ (LLZO) garnets are among the most promising solid electrolytes to be employed in future Li-ion batteries [1]. However, the application of LLZO could be hampered by its reactivity with water and the occurring H^+/Li^+ exchange in its crystal structure [2, 3]. To investigate and understand this phenomenon, quantitative information about the H^+-uptake is of uttermost interest.

In this work, we present method for the determination of H in LLZO garnets using laser induced breakdown spectroscopy (LIBS). Reliable signal quantification was obtained by preparing matrix-matched standards, carefully optimizing the measurement parameters (*e.g.* atmosphere, laser energy, gate delay) and applying appropriate signal normalization as well as background correction. The developed procedure was used to conduct depth-profiling experiments on various LLZO samples, confirming the presence of significant amounts of H in the garnets.

[1] Murugan, Thangadurai & Weppner (2007), *Angew. Chem. Int. Ed.* **46**, 7778-7781. [2] Jin & McGinn (2013), *J. Power Sources* **239**, 326-331. [3] Larraz, Orera & Sanjuán (2013), *J. Mater. Chem. A* **1**, 11419-11428