Sample preparation and analysis of LLZO ceramics for solid state batteries with Cryo FIB/SEM and aberration corrected analytical STEM

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Solid state batteries are promising devices for high capacity energy storage in future electric vehicles. For stable, efficient and long life operation of these batteries stable interfaces of different layers are crucial. To optimize the interface between lithium and the solid electrolyte various interlayer materials are under development. Here we report our cryo-FIB/SEM, HRSTEM and EELS analysis of aluminum doped LLZTO (Lithium Lanthanum Tantalum Zircon Oxide) solid-state electrolyte coated with tin interlayer.

To analyze the morphology and chemical composition of coated LLZTO, FIB (focused ion beam) cross sections were prepared (Zeiss crossbeam 550 scanning electron microscope) and analyzed with EDS (Oxford X-Max 150). In a second step, a several µm thick lamella was prepared by FIB lift out technique [1]. To reduce beam induced damage the sample was finally polished inside the FIB/SEM under cryogenic conditions (-160° C, Quorum PP3010). After finishing a TEM transparent lamella, the sample was warmed up to room temperature and transferred via an argon glove box on a TEM vacuum holder. High resolution analysis of the lamella was performed in a probe corrected JEOL ARM 200F electron microscope equipped with a JEOL Dual EDS system. The Li K line is not accessible in conventional EDS, therefor we used EELS (Electron Energy Loss Spectroscopy, Gatan Quantum ER Spectrometer) to measure the distribution of Lithium.

Results of STEM imaging and EELS mapping are shown in figure 1. STEM imaging with atomic resolution of the aluminum precipitate is shown in figure 2.

[1] Warres C., Lutz T. and Burkhardt C., Insight into LLZTO slid state battery material

[2] Mayo, M. and A.J. Morris, Structure Prediction of Li–Sn and Li–Sb Intermetallics for Lithium-Ion Batteries Anodes. Chemistry of Materials, 2017. 29 (14): p. 5787-5795

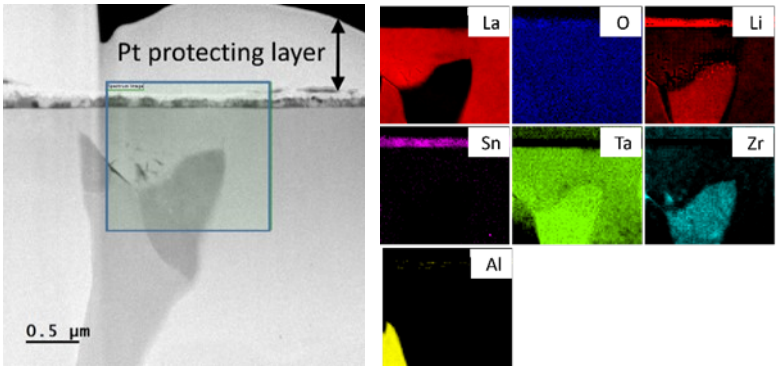

Fig. 1: STEM ADF image and EELS elemental mapping at 200keV in JEOL ARM200F.

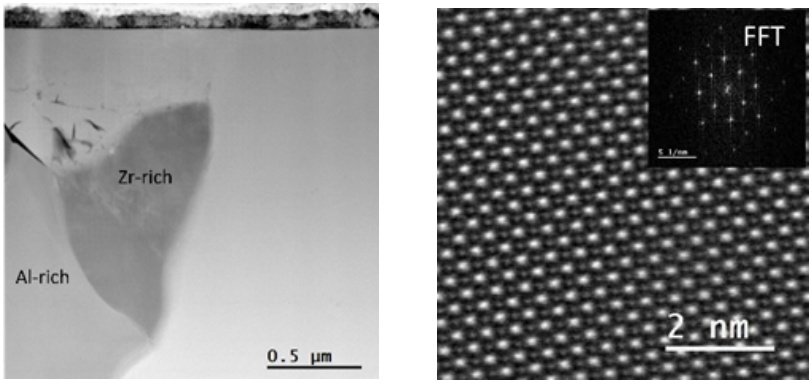

Fig.2: STEM ADF image (left) and atomic resolution image of the aluminum rich precipitate (right).