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The role of $(NO_3)^{2-}$ and Cl^- ions on Sol-Gel synthesis of cubic $Li_7La_3Zr_2O_{12}$ solid electrolytes

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Nowadays, garnet type solid electrolytes are extensively studied electrolytes for Li-ion batteries along with other types of electrolytes such as Perovskite, LISICON and LIPON. Cubic $Li_7La_3Zr_2O_{12}$ (LLZO) phase is relatively recently found composition that shows satisfying ionic conductivity. However, the room temperature stable form of LLZO is tetragonal and either adding stabilizers or increasing sintering temperature have been employed to stabilize cubic phase at room temperature. Recently, it was found that the cubic phase stabilization temperature could be lowered by using solution-based synthesis. However, there is a lack of knowledge on how the anions acting for the stabilization of cubic LLZO. In this work, the effect of $(NO_3)^{2-}$ and Cl^- ions on the cubic phase stabilization was discussed through the sol-gel synthesis. Zr $(NO_3)^2$ and $ZrOCl_2$ were selected as $(NO_3)^{2-}$ and Cl^- anion sources, respectively. 20 mole % Al content as cubic stabilizer was kept constant. After calcination, as-synthesized powders were sintered by using both conventional powder bed sintering and spark plasma sintering (SPS). Crystal structures of calcined powders and sintered pellets were measured by using X-Ray Diffraction (XRD). Combined TGA-FTIR was used to analyze exhaust gases at ppm level. Grain size as well as particle morphology were evaluated by using scanning electron microscope (SEM). True densities and ionic conductivities of sintered pellets were measured by using Archimedes method and AC impedance spectroscopy, respectively. Results showed that high purity cubic LLZO phase in a powder form was obtained after calcination at 1000°C by using $ZrOCl_2$ precursor whereas larger size $(NO_3)^{2-}$ anions inhibit the cubic phase stabilization. Since there were $(NO_3)^{2-}$ anions in the solution that came from $LiNO_3$ and $La(NO_3)^3$, it can be concluded that a limited $(NO_3)^{2-}$ anion concentration was needed to stabilize cubic phase.

Biography

Servet Turan is a Professor at Materials Science and Engineering at the Anadolu University in Eskisehir, Turkey. He has received his BSc in 1988 at Istanbul Technical University and MSc in 1990 at Leeds University whereas, he finished his PhD degree at Cambridge University in 1995. Since then, he has been at Anadolu University. His research interests include the lithium-ion batteries, thermoelectric materials, advanced ceramics and electron microscopy based techniques for characterisation of all sorts of materials including in-situ heating.

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