

Lithium-content-dependent ionic conductivity of NASICON-Type LATP solid electrolytes synthesized by the molten flux method

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Abstract: The article provides a comparative assessment of the structural and electrochemical properties of lithium aluminum titanium phosphate (LATP) solid electrolytes with different lithium contents LATP ($\text{Li}_{1.3-1.5}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$). LATP polycrystals with NASICON-type structure were synthesized by the molten flux method, which included mixing LiNO_3 , Al_2O_3 , TiO_2 , $\text{NH}_4\text{H}_2\text{PO}_4$ and $\text{CO}(\text{NH}_2)_2$ precursors, followed by staged heating at 170°C, 500°C, and 800°C, pressing, and final sintering at 800°C for 4 hours. X-ray diffraction analysis confirmed the formation of the main LATP crystalline phase, while an increase in lithium concentration led to the appearance of secondary phases such as $\text{Li}_4\text{P}_2\text{O}_7$, AlPO_4 , and TiP_2O_7 , which affected ionic conductivity. Electrochemical impedance spectroscopy showed that LATP ($\text{Li}_{1.4}$) exhibits the highest ionic conductivity ($1.66-1.85 \times 10^{-4}$ S/cm at 25°C) compared to $\text{Li}_{1.3}$ and $\text{Li}_{1.5}$ samples due to its higher structural density and optimal phase composition. The temperature dependence analysis revealed that increasing temperature enhances lithium-ion mobility, confirming thermally activated ion transport. Therefore, the $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ composition demonstrates the most promising characteristics as a solid electrolyte for all-solid-state lithium batteries.

Keywords: lithium-ion batteries; NASICON; LATP; RF magnetron sputtering; annealing; electrochemical impedance spectroscopy; electrochemical performance.

1. Introduction

In recent years, the rapid development of portable electronics, electric vehicles, and large-scale energy storage systems has intensified the demand for safer and higher-performance energy storage technologies. Conventional lithium-ion batteries employing liquid electrolytes have achieved remarkable commercial success; however, they still face several limitations related to

safety, thermal stability, electrolyte leakage, and the formation of lithium dendrites during cycling. Consequently, the development of all-solid-state lithium batteries (ASSLBs) has become one of the most actively explored directions in modern electrochemical energy storage research. In ASSLB systems, the flammable liquid electrolyte is replaced by a solid-state electrolyte, which simultaneously serves as an ion-conducting medium and a physical separator between the electrodes.

Solid-state electrolytes (SSE) are a broad class of compounds – oxides (perovskite and garnet types), phosphates (NASICON), sulfides, hydrides, halides, and glasses (borate, phosphate, etc.) – that are being actively studied as an alternative to liquid electrolytes (Waetzig et al., 2020; Zhang et al., 2018). Interest in them is due to a number of advantages: high thermal and electrochemical stability, a wide electrochemical window, and a significant shear modulus that prevents the growth of lithium dendrites (Liu et al., 2018; Yang et al., 2020; Zhu et al., 2015). An additional advantage is safety and environmental friendliness, since SE eliminates the risk of liquid leakage.

Among the different categories of solid electrolytes, oxide-based materials are considered particularly attractive because of their superior chemical stability in air and compatibility with high-voltage cathode materials. Compared to sulfide electrolytes, oxide systems demonstrate better environmental stability and mechanical strength, which are important for practical battery applications. Among oxide electrolytes, NASICON-type lithium conductors have attracted extensive attention due to their unique crystal structure and favorable electrochemical properties. NASICON materials possess a rigid three-dimensional framework that enables fast ion transport through interconnected channels, making them promising candidates for solid-state lithium batteries (Zhang et al., 2023; Moustafa et al., 2020; Wolfenstine et al., 2018; Bachman et al., 2016).

Of the various types of solid electrolytes, materials with a NASICON structure, in particular $\text{Li}_{1.3}\text{Al}_{0.3}\text{Ti}_{1.7}(\text{PO}_4)_3$ (LATP), attract special attention. Their crystal lattice is based on the rhombohedral structure of $\text{LiTi}_2(\text{PO}_4)_3$ (LTP, space group $R\bar{3}c$), formed by a three-dimensional network of octahedral TiO_6 and tetrahedral PO_4 blocks. As illustrated in Figure 1, this architecture creates channels for the migration of Li ions. Substitution of some Ti^{4+} cations with trivalent ions (Al^{3+} , Fe^{3+} , Y^{3+} , etc.) leads to the formation of LATP compounds. The introduction of Al^{3+} increases the concentration of mobile Li ions and weakens the Li-O bond, which contributes to an increase in ionic conductivity (Randau et al., 2020). Given these advantages, LATP materials are considered promising candidates for use in thin-film solid-state electrolytes (Reddy et al., 2020).

The ionic transport mechanism in LATP is associated with the migration of lithium ions through the interconnected NASICON framework. Due to this three-dimensional conduction pathway, LATP materials can exhibit ionic conductivity values reaching 10^{-4} - 10^{-3} S/cm under optimized conditions. In addition, LATP demonstrates good thermal stability and structural robustness, which are essential for long-term electrochemical cycling. However, several challenges still limit the practical implementation of LATP electrolytes. One of the major issues is lithium deficiency arising during synthesis or thin-film deposition processes. Lithium loss at elevated temperatures can lead to the formation of impurity phases, increased grain boundary resistance, and reduced electrochemical performance.

In particular, sputtering has become widespread because it enables precise control of film thickness, composition, and uniformity. This method is especially suitable for thin-film microbattery applications requiring homogeneous electrolyte layers. However, magnetron sputtering is often accompanied by lithium losses during deposition, resulting in deviations from stoichiometry and reduced ionic conductivity of the films (Zhang et al., 2018; Wu et al., 2023; Xiao et al., 2019). To compensate for these losses, LATP compositions with controlled excess lithium were synthesized in this work to maintain optimal electrochemical characteristics of the material (Lovett et al., 2024).

In addition to lithium evaporation during sputtering, the electrochemical performance of LATP thin films strongly depends on the quality of the target pellet material. Parameters such as phase purity,

crystallinity, density, and particle morphology directly influence the properties of the deposited films. Therefore, obtaining homogeneous and highly crystalline LATP ceramics with optimized lithium concentration is essential for successful thin-film fabrication.

The present study focused on obtaining a single-phase, highly conductive LATP material. Among the various synthesis approaches, the molten flux method has attracted significant attention due to its ability to produce well-crystallized materials with controlled morphology and improved ionic conductivity (Nuernberg et al., 2024). This method involves dissolving the precursors in a molten salt medium, which acts as a flux to promote ion diffusion, reduce reaction temperature, and enhance phase purity. Compared to conventional solid-state or sol-gel techniques, the molten flux method offers advantages such as shorter reaction times, uniform particle size distribution, and higher crystallinity (Tolganbek et al., 2021; Mashekova et al., 2022; Baltash et al., 2023).

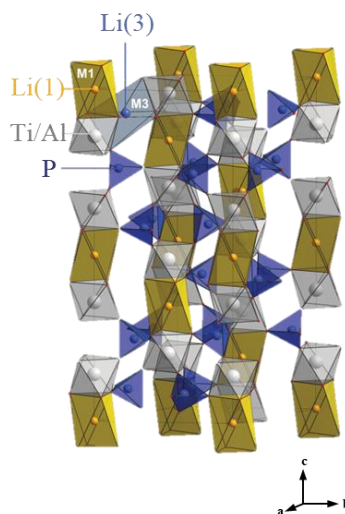


Figure 1. Crystal structure of LATP (DeWees & Wang, 2019)

The molten flux method has demonstrated excellent potential in achieving materials with high ionic conductivity. However, compositional optimization remains necessary, as partial lithium loss may occur during the sputtering process used for thin-film electrolyte fabrication (Arbi et al., 2007). This lithium deficiency can lead to structural instability and reduced ionic performance. Therefore, fine-tuning the target pellet composition is essential to compensate for lithium loss and ensure the formation of a stable, highly conductive LATP phase during thin-film deposition (Liu et al., 2017).

Furthermore, variations in lithium concentration can significantly influence the microstructure, grain connectivity, and grain boundary resistance of LATP ceramics, all of which strongly affect ionic transport properties. Therefore, systematic investigation of lithium content is necessary to determine the optimal composition for achieving both phase stability and enhanced ionic conductivity.

In this work, NASICON-type LATP ceramics with different lithium contents ($x = 0.3-0.5$) were synthesized using the molten flux method to systematically investigate the influence of lithium concentration on phase composition, microstructure, densification, activation energy, and ionic conductivity. Unlike previous studies that mainly focused on conventional LATP compositions or different synthesis routes, this work identifies the optimum lithium composition for producing dense LATP ceramic targets intended for subsequent thin-film electrolyte fabrication by magnetron sputtering.

2. Materials and methods

2.1. Material preparation

NASICON-type LATP ($\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$, $x = 0.3-0.5$) solid electrolytes with various compositions were synthesized using the molten-flux method, as described in studies. To prepare LATP ($x = 0.3-0.5$), precursor powders of LiNO_3 (99 %), Al_2O_3 (99 %), TiO_2 (99.5 %), $\text{NH}_4\text{H}_2\text{PO}_4$ (98 %), and $\text{CO}(\text{NH}_2)_2$ (99.5 %) were accurately weighed and mixed according to the required stoichiometric ratios. An additional 5 % excess of lithium was introduced into all samples during the initial precursor preparation stage to compensate for potential lithium loss occurring throughout the sintering process.

The mixture was preheated at 170 °C for 30 min to remove volatile components and initiate precursor decomposition. The resulting intermediate was finely ground and calcined in a muffle furnace at 500 °C for 5 h with a controlled heating rate of 5 °C/min to promote phase formation.

Following calcination, the powders were further treated at 800 °C for 4 h under the same heating rate and subsequently pressed into 10 mm diameter pellets with pressure of 10 tons (~1.25 GPa). Final densification was achieved by sintering the pellets, covered with mother powders at 800 °C for 4 h in air. For electrochemical measurements, thin gold films (~100 nm) were deposited on both sides of the sintered pellets by magnetron sputtering to serve as blocking electrodes.

2.2. Material characterization

After pellet preparation, their structural, morphological, and electrochemical properties were systematically characterized. The crystal structure was examined by X-ray diffraction (XRD) on a SmartLab (Rigaku, Japan) setup using Cu K α radiation ($\lambda_{\text{CuK}\alpha 1} = 1.54056 \text{ \AA}$, $\lambda_{\text{CuK}\alpha 2} = 1.54439 \text{ \AA}$) in the Bragg-Brentano reflection geometry. The surface morphology of the sintered pellets was observed using scanning electron microscopy (SEM, JEOL JSM- IT800, Japan).

2.3. Electrochemical investigation

The ionic conductivity of pelletized LATP prepared by various compositions was measured by electrochemical impedance spectroscopy (EIS) (potentiostat/galvanostat, Metrohm AutoLab 204, the Netherlands) in the frequency range of 1 Hz-1 MHz with a potential amplitude of 10 mV. Obtained resistance (R) values and parameters such as thickness (d) and area of pellets (S) were used to calculate the ionic conductivity (σ) of the samples by an equation of $\sigma = d/R \times S$. The activation energy for Li^+ conduction was determined from the temperature dependence of the ionic conductivity using Arrhenius plots over the range of 25-85 °C.

3. Results

The obtained results confirmed the successful synthesis of NASICON-type $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (LATP) ceramics with high crystallinity using the molten flux method.

XRD analysis revealed that all samples predominantly consisted of the rhombohedral LATP phase, while only minor secondary phases appeared at higher lithium contents. The diffraction peaks indicated good crystallization and structural stability of the synthesized materials.

SEM observations demonstrated that lithium concentration significantly influenced the morphology and densification of the ceramics. The $\text{Li}_{1.4}$ composition exhibited larger and more densely packed grains with improved intergranular connectivity, whereas the $\text{Li}_{1.5}$ sample showed increased grain heterogeneity. These microstructural differences strongly affected the electrochemical properties of the materials.

EIS confirmed thermally activated lithium-ion transport behavior for all compositions. With increasing temperature, the resistance decreased while ionic conductivity increased, indicating enhanced lithium-ion mobility within the NASICON framework. $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ demonstrated the best electrochemical performance, including the highest density, lowest activation energy, and maximum

ionic conductivity of 1.85×10^{-4} S/cm at 25°C. The improved performance is attributed to optimized lithium content, enhanced grain connectivity, and reduced grain-boundary resistance.

3.1. XRD analysis

The X-ray diffraction (XRD) patterns of the synthesized $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (LATP, $x = 0.3-0.5$) ceramics with different Li_{1+x} contents are shown in Figure 2. All samples exhibit well-defined diffraction peaks corresponding to the characteristic reflections of the NASICON-type rhombohedral structure (space group $R\bar{3}c$), consistent with the standard JCPDS card for $\text{LiTi}_2(\text{PO}_4)_3$ (PDF# 35-0354). The observed diffraction patterns are in good agreement with previous reports on NASICON-type LATP synthesized by molten-flux and solid-state methods (Tolganbek et al., 2021; Baltash et al., 2023; Öksüzöğlu et al., 2024). The sharp and intense peaks confirm the high crystallinity of the obtained materials. For the composition $\text{Li}_{1.3}\text{Al}_{0.3}\text{Ti}_{1.7}(\text{PO}_4)_3$, a pure NASICON phase was obtained without detectable secondary phases. In contrast, when the Al content was increased to $x = 0.4$ and 0.5 , weak additional peaks appeared in the XRD patterns, which were indexed to minor secondary phases such as AlPO_4 , $\text{Li}_4\text{P}_2\text{O}_7$, and TiP_2O_7 . The presence of these phases suggests that excessive Al substitution and lithium volatilization during high-temperature processing may lead to partial decomposition and phase segregation.

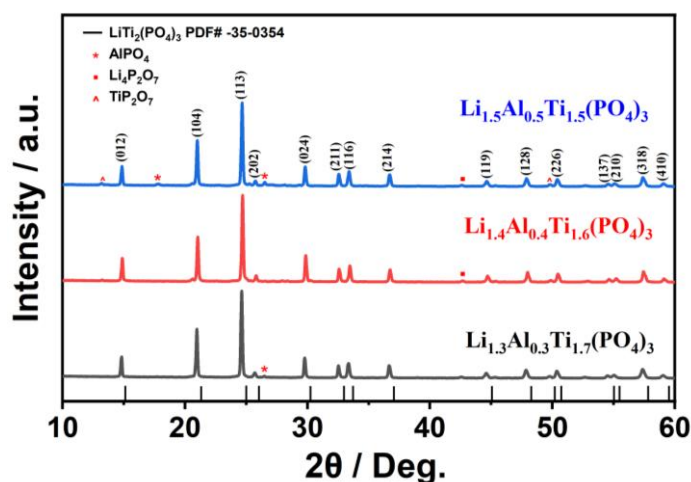


Figure 2. XRD patterns of LATP (a) 0.3, (b) 0.4, (c) 0.5 samples

Nevertheless, the main diffraction peaks remain dominant for all compositions, indicating that the NASICON-type phase is the primary crystalline phase across the studied Li_{1+x} content range ($x=0.3-0.5$). Moreover, with increasing Al substitution, a slight shift of the diffraction peaks toward higher 2θ angles can be observed, which maybe attributed to the partial replacement of larger Ti^{4+} ions ($0.605 \text{ \AA}$) by smaller Al^{3+} ions ($0.535 \text{ \AA}$) in the crystal lattice. This lattice contraction is consistent with previous reports on Al-doped LTP materials. Overall, the XRD results confirm that Al doping successfully stabilizes the NASICON framework while minor secondary phases appear at higher Al contents due to lithium loss and local structural distortions. Reliability factors obtained from Rietveld refinement of the LATP samples are summarized in Table 1.

Table 1. Reliability factors obtained from Rietveld refinement of the LATP samples

Sample	R_{wp} (%)	R_p (%)	S	χ^2	Main phase (wt. %)
Li _{1.3}	12.33	8.69	2.07	4.30	97.8
Li _{1.4}	9.40	7.26	1.59	2.54	88.2
Li _{1.5}	11.10	7.88	1.94	3.75	77.0

To further validate the crystal structure and phase composition, Rietveld refinement was performed for all samples. The calculated diffraction patterns showed good agreement with the experimental data, as indicated by the refinement confirmed that the NASICON-type LATP phase was the dominant crystalline phase in all samples (R_{wp} = 9.40-12.33 %, S = 1.59-2.07). In addition, minor secondary phases were detected, including $AlPO_4$ in Li_{1.3}, $Li_4P_2O_7$ in Li_{1.4}, and $Li_4P_2O_7$, $AlPO_4$, TiP_2O_7 in Li_{1.5}. (Ma et al., 2016; Yang et al., 2021). Table 2 shows the refined lattice parameters of LATP samples obtained from Rietveld refinement.

Table 2. Refined lattice parameters of LATP samples obtained from Rietveld refinement

Sample	Phase	a, Å	b, Å	c, Å	α , °	β , °	γ , °
Li _{1.3}	Lithium aluminum titanium tris(orthophosphate)	8.5067 (4)	8.5067 (4)	20.836 (2)	90.000	90.000	120.000
	Aluminum phosphate	13.494 (13)	13.494 (13)	13.494 (13)	90.000	90.000	90.000
Li _{1.4}	Lithium aluminum titanium tris(orthophosphate)	8.4987 (7)	8.4987 (7)	20.8166 (19)	90.000	90.000	120.000
	Tetralithium diphosphate(V)	8.577 (3)	7.109 (3)	5.183 (2)	111.51 (4)	89.94 (5)	103.005 (19)
Li _{1.5}	Lithium aluminum titanium tris(orthophosphate)	8.5063 (4)	8.5063 (4)	20.8626 (14)	90.000	90.000	120.000
	Tetralithium diphosphate(V)	8.580 (8)	7.142 (10)	5.187 (8)	112.37 (16)	90.31 (12)	103.08 (9)
	Berlinite	4.941 (7)	4.941 (7)	11.06 (3)	90.000	90.000	120.000
	Titanium phosphate	26.11 (16)	26.11 (16)	26.11 (16)	90.000	90.000	90.000

Rietveld refinement confirmed that the LATP phase remained the dominant crystalline phase in all samples, while minor changes in the lattice parameters were observed with increasing lithium content. The refinement also revealed the presence of secondary phosphate phases, particularly for the $\text{Li}_{1.5}$ composition, which is consistent with the XRD analysis.

3.2. Scanning electron microscopy

Figure 3 shows SEM images of the surface of LATP samples with different aluminum contents ($x = 0.3, 0.4$, and 0.5). It is observed that changing the stoichiometry has a significant impact on the morphology of the ceramic materials. For the LATP composition ($x = 0.3$), the microstructure is characterized by relatively fine and uniformly distributed grains of predominantly rectangular shape (Öksüzöğlu et al., 2024). Grain boundary regions are clearly defined, and individual pores indicate incomplete density of the material. With an increase in aluminum content to $x = 0.4$, coarsening of the crystallites with more distinct faceting and the formation of large block grains is observed. The grains are densely packed, indicating more intense crystallite growth and improved microstructural connectivity compared to the sample with $x = 0.3$. For LATP ($x = 0.5$), the characteristic polygonal particle shape is retained, but a broader grain size distribution is observed, including both small and large crystallites. This heterogeneity may be due to changes in grain growth kinetics with increased aluminum concentration.

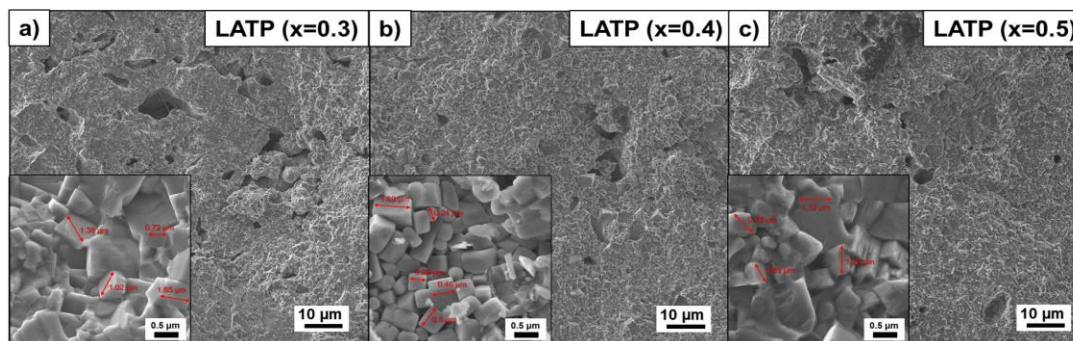


Figure 3. SEM images of $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (a) 0.3, (b) 0.4, (c) 0.5 pellets

In addition, the reduction in visible pore regions for the $x = 0.4$ composition suggests improved densification during the sintering process. Better grain-to-grain contact can facilitate lithium-ion migration by reducing grain-boundary resistance and creating more continuous ion-conduction pathways throughout the ceramic matrix. Such structural characteristics are favorable for achieving enhanced electrochemical performance in NASICON-type solid electrolytes.

For the $x = 0.5$ sample, although grain growth remains pronounced, the coexistence of differently sized crystallites indicates less uniform microstructural evolution. The presence of heterogeneous grains may introduce local structural disorder and nonuniform grain-boundary regions, which can negatively influence ionic transport. Furthermore, excessive aluminum substitution may promote the formation of secondary phases and induce local lattice distortions, leading to reduced structural homogeneity.

$\text{Li}_{1.4}$ composition demonstrated optimized microstructure, high densification, and uniform grain distribution play critical roles in improving lithium-ion transport properties. Therefore, controlling aluminum concentration is essential for tailoring the microstructural and electrochemical characteristics of LATP ceramics intended for solid-state electrolyte applications.

The SEM micrographs reveal that all LATP ceramics consist of densely packed polyhedral grains with well-developed faceted morphology, although noticeable differences in grain size and packing

density are observed with increasing lithium content. The measured grain size ranges from approximately 0.72–1.65 μm for LATP ($x = 0.3$), 0.24–1.69 μm for LATP ($x = 0.4$), and 0.69–1.26 μm for LATP ($x = 0.5$). The $x = 0.3$ sample exhibits relatively large angular grains together with visible intergranular pores, indicating incomplete densification. In contrast, LATP ($x = 0.4$) shows a more homogeneous microstructure with finer grains and a narrower grain size distribution, resulting in improved particle packing and reduced porosity. For LATP ($x = 0.5$), grain growth becomes more pronounced again, with larger faceted grains coexisting with smaller particles, leading to a broader microstructural heterogeneity. These observations suggest that a lithium content of $x = 0.4$ promotes the most uniform grain development and densification, which is consistent with its superior ionic conductivity compared with the other compositions.

Thus, the SEM study demonstrates that increasing the aluminum content promotes grain growth and microstructure densification. However, with a further increase ($x = 0.5$), increased grain heterogeneity is observed, which may affect the ionic conductivity and mechanical properties of the resulting ceramics.

4. Discussion

EIS measurements of LATP pelletized samples ($x = 0.3$ – 0.5) were performed over the temperature range of 25 to 85°C in order to evaluate the ionic transport properties and temperature-dependent electrochemical behavior of the synthesized materials. Figure 4 presents the impedance spectra obtained for the $\text{Li}_{1.3}$, $\text{Li}_{1.4}$, and $\text{Li}_{1.5}$ compositions. All spectra exhibit pronounced semicircular arcs in the high- and intermediate-frequency regions, corresponding to the combined contribution of bulk and grain-boundary resistance within the electrolyte. In the low-frequency region, inclined tails are observed, which are attributed to the polarization effect of the stainless steel blocking electrodes and the accumulation of lithium ions at the electrode/electrolyte interface (Xiao et al., 2023).

To analyze the impedance behavior, the experimental data were fitted using equivalent electrical circuits consisting of bulk resistance (R_b), grain-boundary resistance (R_{gb}), and constant phase elements (CPEs), which account for non-ideal capacitive behavior arising from structural inhomogeneity and grain-boundary effects. The fitting results demonstrated good agreement with the experimental spectra, confirming the validity of the selected equivalent circuit model for describing ionic transport in LATP ceramics.

As the temperature increased from 25 to 85°C, a significant reduction in the diameter of the semicircular arcs was observed for all compositions. This behavior indicates a decrease in total resistance and enhancement of lithium-ion mobility at elevated temperatures. The improved conductivity at higher temperatures can be explained by thermally activated ion transport, where lithium ions gain sufficient energy to migrate more easily through the NASICON framework and across grain boundaries. Such temperature-dependent behavior is characteristic of oxide solid electrolytes and confirms the ionic conduction mechanism in LATP materials.

Among the investigated samples, the $\text{Li}_{1.4}$ composition exhibited the smallest semicircle diameter and therefore the lowest total resistance across the studied temperature range. This result suggests that the $\text{Li}_{1.4}$ sample possesses the most favorable lithium-ion transport pathways and optimal balance between lithium concentration, phase composition, and microstructural characteristics. The enhanced ionic conductivity of this composition may be associated with improved grain connectivity and reduced grain-boundary resistance resulting from the optimized lithium content. In contrast, the $\text{Li}_{1.5}$ composition demonstrated the highest resistance values, indicating less efficient lithium-ion transport. Excess lithium concentration may contribute to the formation of secondary phases, lattice distortion, or increased structural disorder, which can hinder ion migration and increase resistance.

Furthermore, the differences in impedance behavior among the compositions indicate that lithium concentration strongly influences both bulk and interfacial conduction processes in LATP ceramics. Variations in lithium content can affect crystal structure stability, grain growth, and densification, all of which play important roles in determining the electrochemical performance of solid electrolytes. The obtained results therefore confirm that careful optimization of lithium concentration is necessary to achieve high ionic conductivity and stable electrochemical characteristics for thin-film electrolyte applications.

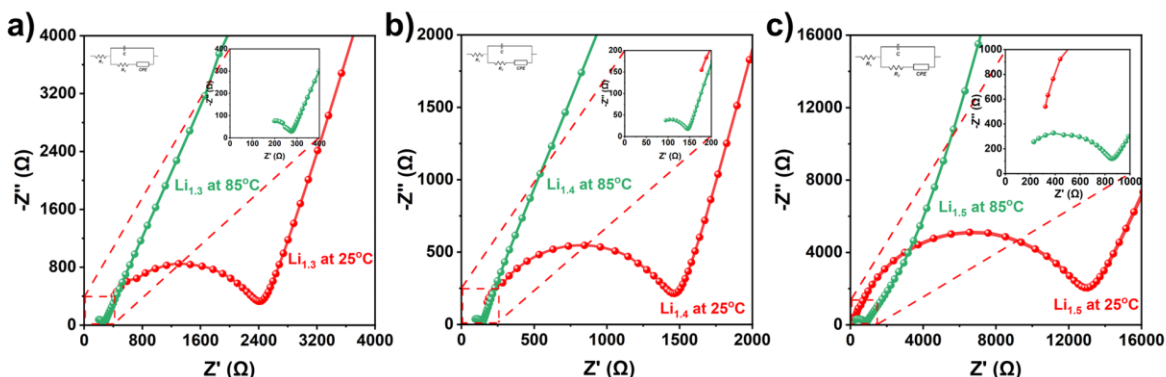


Figure 4. Nyquist plot of $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (a) 0.3, (b) 0.4, (c) 0.5 pellets

Table 3 presents the geometric parameters of the tablets, the sample density, and the calculated ionic conductivity values at 25°C and 85°C. The $\text{Li}_{1.4}$ sample demonstrated the highest conductivity values: $\sigma_{\text{total}} = 1.85 \times 10^{-4}$ S/cm at 25°C and $\sigma_{\text{total}} = 1.45 \times 10^{-3}$ S/cm at 85°C. For comparison, $\text{Li}_{1.3}$ exhibits intermediate values (0.91×10^{-4} S/cm at 25 °C and 0.84×10^{-3} S/cm at 85 °C), while $\text{Li}_{1.5}$ has the lowest conductivity (0.174×10^{-4} S/cm and 0.27×10^{-3} S/cm, respectively). It should be noted that $\text{Li}_{1.4}$ also has the highest density (2.668 g/cm^3), indicating a more densely packed structure and better formation of intergranular contacts, which facilitate efficient ion transport. The obtained ionic conductivity (1.85×10^{-4} S/cm) is comparable to the values reported by Tolganbek et al. (2021) and Baltash et al. (2023), who synthesized LATP using the molten-flux method. However, it remains lower than the highest conductivities reported for highly densified LATP prepared by optimized solid-state routes. This difference may be attributed to variations in sintering conditions, relative density, and secondary phase formation.

Table 3. Characteristics of $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ ($x=0.3-0.5$) pellets

LATP ($x=0.3-0.5$)	d, cm	h, cm	S, cm^2	ρ , g/cm^3	$\sigma_{\text{total}}(25^\circ\text{C})$, $\times 10^{-4}$, S/cm	$\sigma_{\text{total}}(85^\circ\text{C})$, $\times 10^{-3}$, S/cm	E_a , eV
$\text{Li}_{1.3}$	0.772	0.109	0.468	2.57	0.91	0.84	0.36
$\text{Li}_{1.4}$	0.768	0.099	0.463	2.67	1.85	1.45	0.32
$\text{Li}_{1.5}$	0.767	0.111	0.462	2.64	0.17	0.27	0.43

Although the $\text{Li}_{1.4}$ sample contains a lower fraction of the LATP phase than $\text{Li}_{1.3}$, it exhibits the highest ionic conductivity. This behavior suggests that ionic transport is influenced not only by the LATP

phase fraction but also by the lower activation energy, favorable microstructure, and the nature of the secondary phases. The insulating AlPO_4 phase in $\text{Li}_{1.3}$ may contribute to higher grain-boundary resistance, whereas the $\text{Li}_4\text{P}_2\text{O}_7$ phase in $\text{Li}_{1.4}$ has a less pronounced influence on Li^+ transport.

The temperature dependence of conductivity in Arrhenius coordinates confirms the impedance spectroscopy data. The Arrhenius plots of the samples are illustrated in Figure 5. Linear behavior is observed for all compositions, indicating a thermally activated mechanism of lithium-ion transport within the NASICON framework. The good linearity of the curves suggests that lithium-ion conduction occurs through a stable hopping mechanism without significant phase transitions in the investigated temperature range.

Approximation of the experimental curves allowed the determination of the activation energies for ionic conduction. For the $\text{Li}_{1.3}$ composition, the activation energy is 0.36 eV, for $\text{Li}_{1.4}$ it is 0.32 eV, while for $\text{Li}_{1.5}$ it reaches 0.43 eV. The lowest activation energy was recorded for $\text{Li}_{1.4}$, which corresponds to the highest ion mobility and is consistent with its minimum resistance and maximum conductivity values obtained from impedance measurements. The reduced activation energy indicates that lithium ions require less energy to migrate through the crystal lattice and grain boundaries, resulting in more efficient ionic transport.

The enhanced electrochemical performance of the $\text{Li}_{1.4}$ sample can be associated with its optimized lithium concentration and improved microstructural properties. Better grain connectivity and increased densification likely contribute to the formation of continuous lithium-ion transport pathways and reduced grain-boundary resistance. In addition, the relatively uniform grain structure observed in SEM images supports the improved ionic mobility of this composition.

In contrast, the $\text{Li}_{1.5}$ sample is characterized by the highest activation energy and the lowest ionic conductivity. The increase in activation energy suggests more difficult lithium-ion migration due to structural disorder, grain heterogeneity, or the presence of secondary phases formed at excessive lithium content. Such factors can obstruct lithium-ion diffusion pathways and increase interfacial resistance within the ceramic electrolyte.

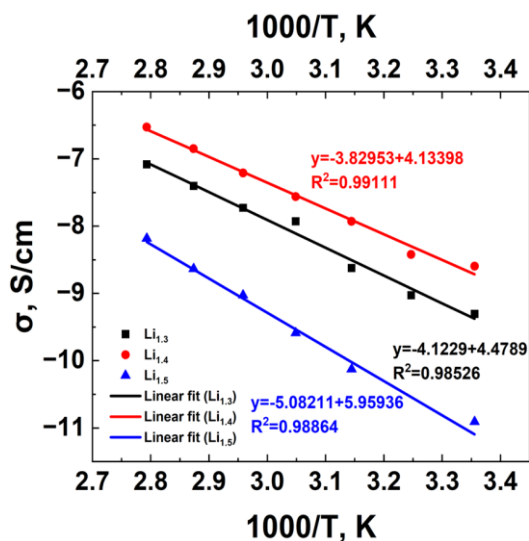


Figure 5. Arrhenius plot of $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (a) 0.3, (b) 0.4, (c) 0.5 pellets

Thus, a combined analysis of the impedance spectra, temperature dependences, and experimental data in the table confirms that the $\text{Li}_{1.4}$ composition is optimal among those studied. It combines

maximum density, minimum resistance, lowest activation energy, and highest ionic conductivity, making it a promising candidate for the further production of thin-film electrolytes.

5. Conclusion

The LATP solid electrolyte synthesis method using melting with flux technology produces polycrystals with a high degree of crystallinity and a dense NASICON-type structure. These samples are characterized by fewer structural distortions and higher ionic conductivity compared to materials obtained by other synthesis methods. The most optimal electrochemical parameters were demonstrated by the $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ sample, which has the highest density and ionic conductivity (up to 1.85×10^{-4} S/cm at 25°C).

The enhanced electrochemical performance of the $\text{Li}_{1.4}$ composition can be attributed to the optimized lithium content, which promotes efficient lithium-ion migration through the NASICON framework while minimizing grain-boundary resistance. In addition, the improved densification and phase purity of this sample contribute to better particle connectivity and reduced structural defects. These results indicate that controlled lithium excess is an effective strategy for obtaining highly conductive LATP electrolytes suitable for thin-film solid-state battery applications.

Structural and morphological analyses confirmed that lithium concentration strongly influences phase formation, grain growth, and densification behavior of LATP ceramics. The $\text{Li}_{1.4}$ sample exhibited the most favorable combination of structural homogeneity, dense microstructure, low activation energy, and high ionic conductivity. In contrast, excessive lithium content resulted in increased grain heterogeneity and reduced electrochemical performance. Overall, the obtained results demonstrate that optimization of lithium concentration is essential for improving the structural stability and ionic transport properties of LATP electrolytes, making the $\text{Li}_{1.4}$ composition a promising candidate for future thin-film all-solid-state lithium battery applications.

Furthermore, the impedance spectroscopy results confirmed that lithium-ion transport in LATP ceramics follows a thermally activated conduction mechanism, with conductivity increasing significantly at elevated temperatures. The combination of high crystallinity, reduced grain-boundary resistance, and improved microstructural connectivity observed for the $\text{Li}_{1.4}$ composition contributed to enhanced ionic mobility throughout the NASICON framework. The molten flux synthesis approach also proved to be an effective method for obtaining homogeneous LATP ceramics with stable electrochemical characteristics. Therefore, the developed $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ material can be considered a promising electrolyte candidate for further fabrication of thin-film solid-state lithium batteries and related electrochemical energy storage systems.

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7. Author Contributions

Conceptualization - A.B., M.Y., Z.Ba.; methodology - A.B., M.Y., Z.Ba.; formal analysis - A.B., M.Y.; investigation - M.Y.; data curation - M.Y., G.T., A.N., A.M.; writing-original draft - A.B.; writing - review & editing - A.B., M.Y., Z.Ba.; visualization - A.B., M.Y., Z.Ba.; supervision - G.N., A.N., Z.Bk., A.M.; project administration - A.N., A.M.

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Балқытылған флюс әдісімен синтезделген NASICON типті LATP қатты электролиттерінің литий мөлшеріне тәуелді иондық өткізгіштігі

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Аңдатпа. Бұл мақалада әртүрлі литий мөлшеріне ие литий алюминий титан фосфатының (LATP) қатты электролиттерінің құрылымдық және электрохимиялық қасиеттеріне салыстырмалы талдау жүргізілді LATP ($\text{Li}_{1.3-1.5}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$). NASICON құрылымды LATP поликристалдары балқытылған флюс әдісі арқылы синтезделді. Синтез процесі LiNO_3 , Al_2O_3 , TiO_2 , $\text{NH}_4\text{H}_2\text{PO}_4$ және $\text{CO}(\text{NH}_2)_2$ прекурсорларын араластыруды, кейін 170°C, 500°C және 800°C температураларда кезең-кезеңімен қыздыруды, престоуді және соңғы 800°C температурада 4 сағат бойы күйдіруді қамтыды. Рентгендік дифракциялық талдау негізгі LATP кристалдық фазасының түзілгенін растады. Сонымен қатар, литий концентрациясының артуы $\text{Li}_4\text{P}_2\text{O}_7$, AlPO_4 және TiP_2O_7 секілді қосымша фазалардың пайда болуына әкеліп, иондық өткізгіштікке әсер ететіні анықталды. Электрохимиялық импеданстық спектроскопия нәтижелері бойынша LATP ($\text{Li}_{1.4}$) үлгісі жоғары құрылымдық тығыздық пен оңтайлы фазалық құрамның арқасында $\text{Li}_{1.3}$ және $\text{Li}_{1.5}$ үлгілерімен салыстырғанда ең жоғары иондық өткізгіштікке ие болды (25°C-та $1.66-1.85 \times 10^{-4} \text{ S/cm}$). Температуралық тәуелділік талдауы температураның жоғарылауы литий иондарының қозғалғыштығын арттыратынын көрсетті, бұл ион тасымалының термиялық белсендірілген табиғатын дәлелдейді. Осылайша, $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ құрамы қатты күйлі литий батареяларына арналған қатты электролит ретінде ең перспективалы қасиеттерді көрсетті.

Түйін сөздер: литий-ионды батареялар; NASICON; LATP; РЖ магнетронды тозаңдату; күйдіру; электрохимиялық импеданстық спектроскопия; электрохимиялық сипаттамалар.

Влияние содержания лития на ионную проводимость NASICON-подобных твердых электролитов LATP, синтезированных методом расплавленного флюса

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Аннотация. В статье представлена сравнительная оценка структурных и электрохимических свойств твердых электролитов на основе литий-алюмо-титан-фосфата (LATP) с различным содержанием лития LATP ($\text{Li}_{1.3-1.5}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$). Поликристаллы LATP с NASICON-подобной структурой были синтезированы методом расплавленного флюса, включающим смешивание прекурсоров LiNO_3 , Al_2O_3 , TiO_2 , $\text{NH}_4\text{H}_2\text{PO}_4$ и $\text{CO}(\text{NH}_2)_2$ с последующим ступенчатым нагревом при 170°C , 500°C и 800°C , прессованием и финальным спеканием при 800°C в течение 4 часов. Рентгенофазовый анализ подтвердил формирование основной кристаллической фазы LATP, тогда как увеличение концентрации лития приводило к появлению вторичных фаз, таких как $\text{Li}_4\text{P}_2\text{O}_7$, AlPO_4 , и TiP_2O_7 , что оказывало влияние на ионную проводимость. Исследования методом электрохимической импедансной спектроскопии показали, что образец LATP ($\text{Li}_{1.4}$) обладает наивысшей ионной проводимостью ($1.66\text{--}1.85 \times 10^{-4} \text{ S/cm}$ при 25°C) по сравнению с образцами $\text{Li}_{1.3}$ и $\text{Li}_{1.5}$ благодаря более высокой структурной плотности и оптимальному фазовому составу. Анализ температурной зависимости показал, что повышение температуры способствует увеличению подвижности литиевых ионов, подтверждая термоактивированный характер ионного транспорта. Таким образом, состав $\text{Li}_{1.4}\text{Al}_{0.4}\text{Ti}_{1.6}(\text{PO}_4)_3$ демонстрирует наиболее перспективные характеристики в качестве твердого электролита для полностью твердотельных литиевых батарей.

Ключевые слова: литий-ионные батареи; NASICON; LATP; РЧ магнетронное распыление; отжиг; электрохимическая импедансная спектроскопия; электрохимические характеристики.