

Structural and Dielectric study of lithium Titanate (LTO) as an alternate anode

A DISSERTATION REPORT

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**MASTERS OF SCIENCE
IN
PHYSICS**

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CANDIDATE'S DECLARATION

We, AKANKSHA SINGH, Roll No(s). 23/MSCPHY/61 and DHRUV MEHTA, 23/MSCPHY/83, students of M.Sc. Physics hereby declares that the project Dissertation titled "Structural and Dielectric study of lithium Titanate (LTO) as an alternate anode" Which is submitted by us to the Department of Applied Physics, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma Associateship, Fellowship or other similar title or recognition.

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DECLARATION

We, **Akanksha Singh (23/MSCPHY/61)** and **Dhruv Mehta (23/MSCPHY/83)** hereby certify that the work which is presented in the Major Project-II titled “Structural and Dielectric study of lithium Titanate (LTO) as an alternate anode” for Li-ion Battery in fulfillment of the requirement for the award of the Master in Science in **Physics** and submitted to the Department of **Applied Physics**, Delhi Technological University, Delhi is an authentic record of our own, carried out during a period from January 2025 to May 2025 under the supervision of Dr. Amrish K. Panwar.

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ABSTRACT

The lithium-ion battery represents one of the most significant technological advancements in energy storage, powering everything from portable electronics to electric vehicles. Among the various components of a Li-ion battery, the anode plays a crucial role in determining its overall capacity, efficiency, and cycle life. Since, the conventional graphitic anodes are widely used. The alternative materials such as silicon, metal oxides, and lithium titanate ($\text{Li}_4\text{Ti}_5\text{O}_{12}$, LTO) are being explored due to their superior electrochemical characteristics. Among these, LTO has gained attention due to its excellent safety, long cycle stability, and fast charging capability. In this research work, LTO has been synthesized by the conventional solid-state method. XRD confirmed the formation of a spinel structure with a space group Fd-3m . The Reitveld Refinement has been conducted on the observed XRD patterns of ($\text{Li}_4\text{Ti}_5\text{O}_{12}$, LTO) to extract critical structural parameters relevant to its application in energy storage. The impedance and permittivity analysis are conducted on the sample using Impedance spectroscopy, revealing that the relaxation process is predominantly controlled by grain boundaries, highlighting their impact on the material's conductivity. Hence, the investigation provides deeper insights into the influence of grain boundaries and dielectric behavior on LTO's performance, contributing to a more comprehensive understanding of its functional properties in lithium-ion battery applications.

Keywords: Lithium-ion batteries, Anode material, LTO

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CHAPTER 1

INTRODUCTION AND OBJECTIVES

1.1 INTRODUCTION

1.1 Background and Motivation

Over the past two decades, lithium titanate spinel ($\text{Li}_4\text{Ti}_5\text{O}_{12}$, LTO) has gained significant attention as an advanced anode formulation for advanced Li-ion batteries owing to its virtually “zero-strain” intercalation behavior, excellent cycle life, and inherent safety versus graphite anodes. Unlike conventional graphite, which undergoes significant volume changes ($\approx 10\%$) during lithiation/delithiation, the cubic spinel framework of LTO (space group $\text{Fd-}3\text{m}$) accommodates Li^+ insertion with negligible lattice distortion $\leq 0.2\%$ at $\sim 1.55\text{ V}$ vs. Li/Li^+ . This remarkable structural stability suppresses the formation of solid-electrolyte interphase (SEI) layers and mitigates capacity fade over hundreds to thousands of cycles. However, pristine LTO suffers from extremely low electronic conductivity ($\sim 10^{-12}\text{ S}\cdot\text{cm}^{-1}$) and relatively hindered Li^+ diffusion kinetics which restrict its high-rate performance and overall energy density.

To address these shortcomings, researchers have explored nano scaling, carbon-coating, doping, and hybrid composites. Yet, a fundamental understanding of how intrinsic microstructure—grain size, porosity, crystallographic strain—and interfacial phenomena govern the electrical and dielectric properties of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ remains incomplete. Deconvoluting the contributions from grain interiors, grain

boundaries, and electrode–electrolyte interfaces under varying temperature and frequency conditions is critical for rational design of high-power LTO electrodes.

1.2 Complex Impedance Spectroscopy and Dielectric Analysis

Complex impedance spectroscopy (CIS) has become the tool of choice to probe the frequency-dependent electrical response of ceramic electrodes and to extract equivalent-circuit parameters (bulk resistance, grain-boundary capacitance, Warburg diffusion, etc.). Likewise, dielectric analysis—measuring the real and imaginary parts of permittivity (ϵ' and ϵ'')—provides insight into dipolar relaxation, space-charge polarization, and AC conductivity mechanisms within the material. Together, these complementary techniques yield a comprehensive picture of charge-transport pathways and energy-dissipation processes in $\text{Li}_4\text{Ti}_5\text{O}_{12}$.

Despite numerous impedance studies on carbon-coated or doped LTO, systematic investigations that correlate Rietveld-refined structural parameters (lattice constants, microstrain, crystallite size) with quantitative impedance/dielectric metrics are scarce. Such correlations are essential for pinpointing the microstructural levers—such as optimized grain-boundary chemistry or controlled defect populations—that enhance high-rate kinetics without compromising cycle stability.

1.3 Scope and Objectives

The central objective of this dissertation is to synthesize phase-pure spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$, perform detailed Rietveld refinement of its X-ray diffraction (XRD) patterns, and

conduct comprehensive complex impedance and dielectric spectroscopy over a broad frequency (10 Hz–1 MHz) at 800°C. Specific objectives include:

Structural Characterization: Confirm spinel phase purity via XRD; refine lattice parameters, atomic positions, crystallite size, and microstrain using Rietveld analysis.

Impedance Spectroscopy: Measure and model Nyquist and Bode plots to extract bulk and grain-boundary resistances, capacitances, and Warburg elements; evaluate activation energies for bulk and interfacial charge-transport processes.

Dielectric Analysis: Determine frequency- and temperature-dependent permittivity (ϵ') and dielectric loss (ϵ'').

Refinement: Finding the values for Crystallite size and microstrain

LITHIUM-ION BATTERY

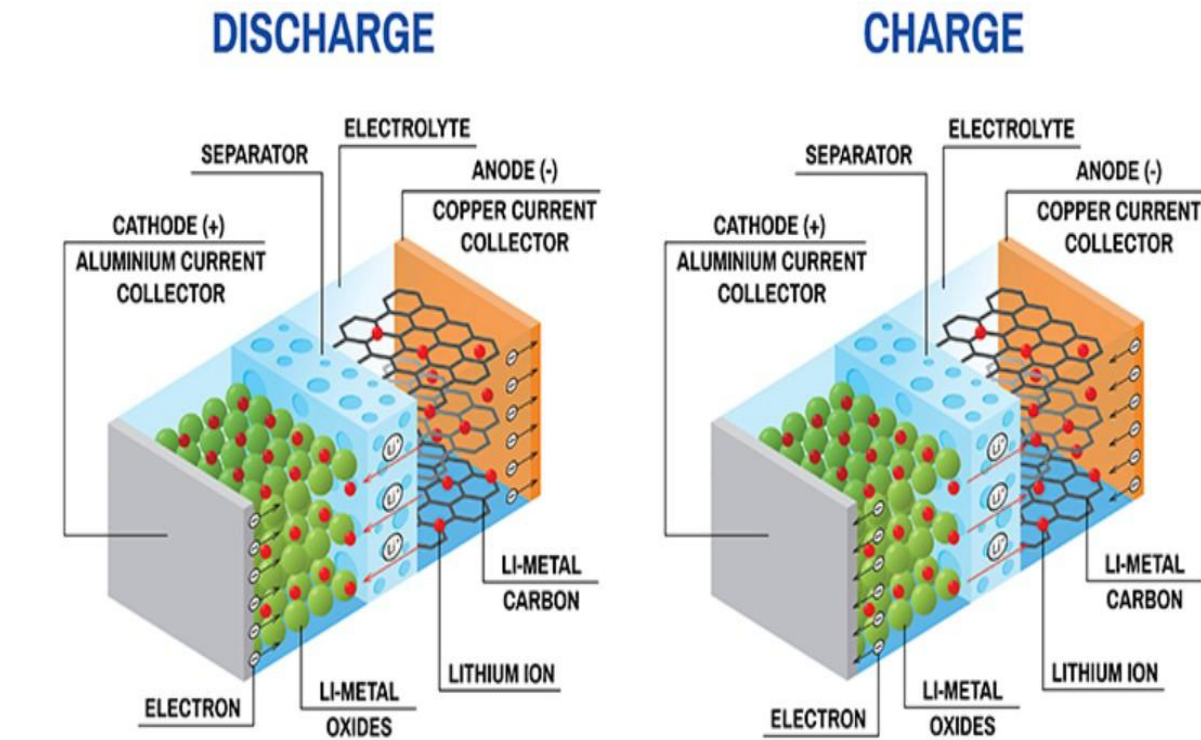


Fig. 1 Lithium-ion Battery Charging and Discharging

Lithium-ion cells underpin today's energy storage solutions because they combine remarkable gravimetric and volumetric energy capacities with lengthy charge–discharge lifespans and minimal standby losses. As a result, these cells have become ubiquitous in portable electronics and are increasingly driving advancements in electric mobility. Beyond consumer and automotive applications, they are also being deployed at the megawatt scale to buffer intermittent renewables and bolster power-grid resilience. However, the widespread deployment of LIBs across diverse sectors necessitates careful optimization of multiple performance parameters,

including energy and power density, charge/discharge cycle life, economic feasibility, operational safety, and environmental sustainability. These performance metrics often involve trade-offs; for instance, improving energy density may compromise safety or lifespan, while reducing costs might affect overall efficiency. These interdependent trade-offs are rooted in the fundamental materials chemistry of the battery components, particularly the electrodes and electrolytes.

Traditional LIB technology primarily employs insertion-type electrodes, such as layered lithium cobalt oxide (LiCoO_2) for the cathode and graphite for the anode, in conjunction with organic liquid electrolytes. While this configuration has proven effective for consumer electronics, it poses limitations in terms of achievable energy density and thermal stability, especially under high-rate or high-temperature operating conditions. To address these limitations, significant research efforts are focused on developing next-generation electrode materials. These include not only advanced insertion-type materials but also conversion-type materials, which offer potentially higher capacities by leveraging different electrochemical mechanisms.

Parallel to electrode innovations, solid-state electrolytes are being investigated as a means to enhance battery safety and enable the use of high-capacity lithium metal anodes. Solid-state batteries promise to overcome the flammability and leakage issues associated with liquid electrolytes, while also supporting higher energy densities and longer lifespans. Nonetheless, challenges such as interfacial stability, ionic conductivity, and manufacturability remain critical hurdles to commercial viability.

LITERATURE REVIEW

■ Li-ion battery

Li-ion batteries are rechargeable electrochemical storage devices that have become indispensable across a variety of fields—from handheld electronics and electric vehicles to utility-scale renewable energy buffering. Their rapid rise in deployment owes much to their excellent specific energy, long-term cycling stability, and lightweight construction, which together permit highly compact and efficient power modules. At the heart of their operation lies the reversible insertion (intercalation) and removal (deintercalation) of lithium ions into the crystal lattices of the anode and cathode materials during charging and discharging cycles.

In a conventional lithium-ion cell, the negative electrode is typically made from layered graphite, whereas the positive electrode employs a lithium–transition-metal oxide—for example, LiCoO_2 . When the battery is charged, lithium ions are drawn out of the cathode, traverse the electrolyte, and become inserted into the graphite’s interlayer galleries. At the same time, electrons travel through the external circuit to maintain charge balance. During discharge, this sequence is simply reversed: lithium ions exit the graphite, migrate back into the metal-oxide lattice, and the liberated electrons flow through the load, delivering usable electrical power. It is this fully reversible shuttling of ions and electrons that underpins the high efficiency, long cycle life, and broad applicability of lithium-ion batteries.

LIBs offer advantages such as low self-discharge and high voltage per cell, but challenges include safety risks like thermal runaway, resource scarcity, and

environmental concerns from production and disposal. Advances in materials, such as silicon anodes and solid electrolytes, aim to enhance their performance, safety, and sustainability.

■ Components of Li-ion Battery

A lithium-ion battery (LIB) comprises several fundamental components that function synergistically to enable efficient energy storage and delivery. These core elements include the anode, cathode, electrolyte, separator, and current collectors. Each component contributes significantly to the battery's electrochemical characteristics, operational safety, and lifespan of the battery system.

Anode:

- This electrode, known as the anode and usually fabricated from graphite, hosts lithium ions throughout the charging process.
- Emerging materials like silicon or lithium titanium oxide (LTO) are used for higher capacity or improved safety.

Cathode:

- The positive electrode typically consists of lithium-rich transition-metal oxides, for example LiCoO_2 or NMC.
- It serves as the source of lithium ions and determines energy density.

Electrolyte:

- A lithium-ion-conductive medium, usually a liquid organic solvent containing lithium salts (e.g., LiPF₆).
- Solid electrolytes are being developed to enhance safety and prevent leakage.

Separator:

- A thin, porous membrane (often polymer-based) that physically isolates the anode from the cathode while permitting the selective passage of lithium ions.
- It prevents short circuits within the cell.

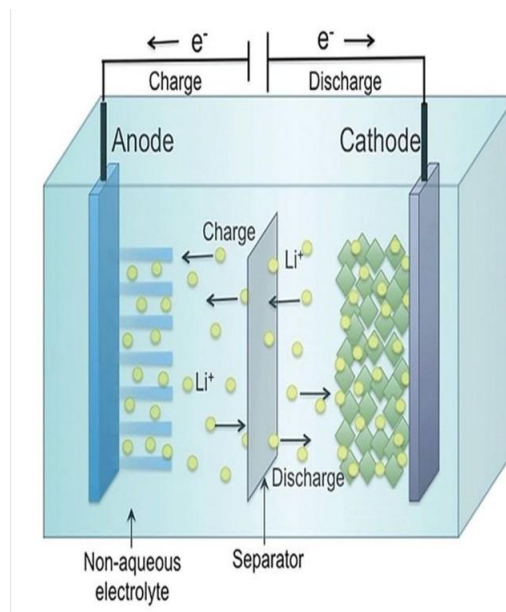


Fig. 2 Working of a Li-ion Battery

1.3.1 LIB working principle

Lithium-ion batteries (LIBs) are primarily comprised of four key elements: the positive and negative electrodes, separator, and electrolyte. The positive and negative electrode

serve as the active materials responsible for the storage and release of electrical energy through reversible lithium-ion intercalation and deintercalation processes. The separator acts as an insulating layer between the anode and cathode, preventing internal short-circuits while still permitting lithium ions to migrate. The electrolyte, in turn, supports ionic conduction by facilitating the movement of Li^+ through the cell. The integrity and coordinated function of these components are

critical to the overall performance and safety of LIBs. Any malfunction—whether in isolation or in combination—can significantly compromise battery safety. During operation, electrochemical reactions within the electrodes generate heat. Under failure conditions, this heat may become excessive and uncontrollable. In particular, damage to the separator can lead to direct contact between the electrodes, triggering a transition from regulated to uncontrolled electrochemical reactions, often accompanied by rapid heat release. In such scenarios, the electrolyte can act as a combustible medium, intensifying thermal runaway.

Therefore, stringent safety protocols and design strategies are essential to mitigate these risks. These protocols must be informed by a thorough understanding of both the controllable and uncontrollable factors contributing to LIB failure mechanisms. When a cell is charged, Li^+ ions are withdrawn from the cathode—typically lithium cobalt oxide (LiCoO_2)—and travel through the electrolyte and separator pores to embed themselves in the graphite anode. Concurrently, electrons move through the external circuit to uphold charge balance. Upon discharge, this sequence inverts: lithium ions migrate back into the cathode host structure, and the electrons' return through the circuit provides electrical power to the connected device.

1.3.2 ANODE MATERIALS

In early battery prototypes, pure lithium metal served as the anode because of its exceptional theoretical capacity and minimal electrochemical potential. However, repeated charging cycles caused lithium to deposit preferentially along the most direct route between electrodes. This uneven growth intensified surface defects, resulting in the gradual emergence of lithium filaments—needle-like protrusions capable of breaching the separator. Over time, these filaments may span the electrolyte and contact the cathode, leading to internal short circuits. In extreme cases, such events can initiate thermal runaway and even spark fires or explosions.

To address these critical safety concerns, researchers have shifted focus toward alternative anode materials that offer improved structural stability and reduced risk of dendrite formation. Three primary categories of such materials have been extensively explored: intercalation materials, which allow reversible insertion of lithium ions into layered or tunneled host structures (e.g., graphite); conversion-type materials, which undergo a complete transformation into new phases during lithium uptake; and alloying materials, which form lithium-rich alloys through electrochemical reactions (e.g., silicon or tin-based anodes). Each of these alternatives presents unique advantages and challenges, and they continue to be central to development of next-generation li-ion batteries.

1.4 TYPES OF ANODE MATERIALS

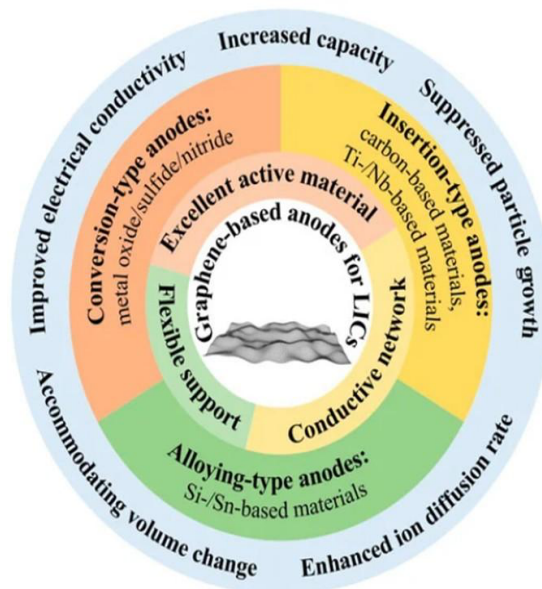


Fig. 3 Types of Anode Materials

1.5 Intercalation materials:

The intercalation mechanism is based on the reversible uptake of Li^+ into the vacant gaps of a host framework, occurring without major distortion of its lattice. Because it does not require breaking or forming covalent bonds, this approach allows for smooth and durable cycling of lithium ions during charge and discharge. Graphite was the pioneering intercalation anode brought to market—first commercialized by Sony in 1991 owing to its low operating potential (~ 0.125 V vs. Li/Li^+) and suppressed dendrite formation.

Graphite continues to be the predominant anode material in lithium-ion batteries, largely attributed to its excellent electrochemical characteristics, abundant availability, and cost-effectiveness. In contrast, lithium titanate ($\text{Li}_4\text{Ti}_5\text{O}_{12}$ or LTO) has been introduced as a potential alternative anode, functioning at a relatively higher operating voltage of around 1.55 V versus Li^+/Li . This elevated voltage improves operational

safety by significantly reducing the risk of lithium plating. While this higher potential further enhances safety by virtually eliminating lithium plating, LTO suffers from a lower specific capacity (175 mAh/g) in comparison to graphite (372 mAh/g), which limits its energy density. Additionally, economic and supply considerations continue to favor graphite over LTO in large-scale applications.

Despite the structural stability and safety benefits of intercalation materials, their relatively low lithium-to-host material ratio results in modest gravimetric capacities compared to other anode technologies, such as conversion and alloying materials. Nonetheless, their reliable performance and maturity make intercalation compounds—particularly graphite—the prevailing choice for current-generation lithium-ion batteries.

1.6 Alloying type materials:

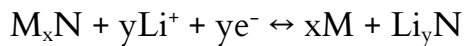
Alloying-type materials represent a prominent class of alternatives to conventional graphite anodes. These materials, which form alloys with lithium, have attracted significant attention due to their substantially higher theoretical gravimetric capacities. Elements such as tin (Sn), silicon (Si), aluminum (Al), and magnesium (Mg) are among the most extensively investigated candidates. Silicon stands out due to its natural abundance and its exceptionally high theoretical capacity of approximately 3600 mAh/g, that is approx. ten times greater than that of graphite (372 mAh/g)[1].

Despite these advantages, alloying materials also exhibit notable challenges that hinder their practical application. The lithiation and delithiation processes induce significant volumetric expansion and contraction — up to ~300% [1] in the case of silicon. These drastic volume fluctuations compromise the mechanical integrity of the anode, leading to structural degradation and loss of electronic connectivity due to mechanical fatigue. Additionally, the incomplete formation of a stable solid electrolyte interphase (SEI) layer, caused by continuous volume changes, results in persistent

electrolyte decomposition and capacity fading. These interfacial issues are further elaborated in the section discussing the electrode–electrolyte interface.

1.7 Conversion type material:

Conversion materials function through an exchange reaction as expressed in equation:



where M is a transition metal (Fe, Co, Mn, etc) and N is an electronegative element (O, S, P, etc). Fe oxides and related transition-metal oxides have attracted considerable interest as negative-electrode materials because they can deliver very high capacity at a modest cost. During lithiation, these materials are fully reduced to their elemental metal forms while generating Li_2O , and the process reverses upon delithiation.

Despite their promising attributes, conversion materials share several challenges with alloying-type anodes. Chief among these is the substantial volume change during cycling, which can compromise structural integrity and contribute to ongoing electrolyte decomposition. Moreover, many conversion-type anodes exhibit inherently slow electrochemical kinetics, leading to significant voltage hysteresis during charge and discharge processes. This pronounced hysteresis reduces the overall energy efficiency of the battery by increasing internal energy losses, thereby limiting their practical viability in high-performance applications.

1.8 Spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as an anode

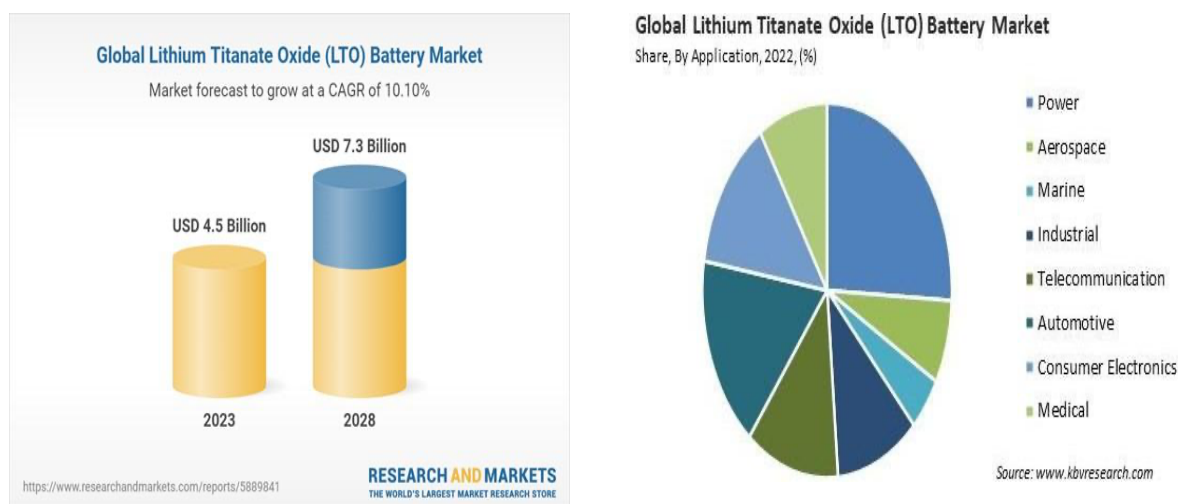


Fig. 4 Global Market of Li-ion Battery with LTO as Anode

Graphite, the conventional anode material in lithium-ion batteries (LIBs), presents certain limitations due to its low electrochemical potential for lithium intercalation. Under specific conditions—such as low temperatures or high current densities—graphite-based anodes can promote lithium plating and dendritic growth, posing significant safety risks including short-circuiting and thermal runaway. To address these challenges, lithium titanate ($\text{Li}_4\text{Ti}_5\text{O}_{12}$, LTO) has been proposed as a promising alternate option for high-power applications. With a higher operating potential of approx. 1.55 V vs. Li/Li^+ , LTO effectively mitigates the formation of lithium dendrites. [2]

One of LTO's most advantageous properties is its "zero-strain" behavior: both lithiated and delithiated phases share identical lattice parameters, resulting in negligible volumetric change $< 0.2\%$ during cycling [3]. This characteristic sharply contrasts with graphite,

which undergoes about a 10% volume expansion that compromises the integrity of the solid electrolyte interphase (SEI) and accelerates electrolyte degradation—a known long-term failure mechanism in conventional LIBs. The mechanical stability of LTO enables extended cycle life without structural fatigue or SEI cracking. Moreover, LTO is composed of relatively abundant elements, supporting scalable production at reduced costs.

Despite its favorable electrochemical and structural properties, the SEI behavior on LTO remains under debate. While its elevated potential theoretically precludes SEI formation, experimental evidence has been inconclusive. Some studies report the presence of surface films, attributing them to cathode-derived oxidation products or electrolyte decomposition, while others suggest the absence of a traditional SEI altogether.

Despite its safety and cycling advantages, LTO incurs a roughly 1.4 V voltage penalty versus graphite, which translates into lower cell-level energy density. To mitigate this loss, LTO is typically paired with high-voltage cathodes. Notable examples include spinel LiMn_2O_4 (LMO), nickel stabilized $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ (LMNO), and layered $\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$ (NMC) materials. These manganese-rich oxides combine elevated operating voltages with the benefits of manganese's abundance and low environmental impact, making them strong candidates for large-scale battery applications.

Nevertheless, transition metal dissolution, particularly manganese, poses a major challenge. The dissolution is believed to be catalyzed by the dominantly adopted electrolyte salt LiPF_6 , which decomposes to form hydrofluoric acid (HF). HF subsequently attacks the cathode material, resulting in accelerated degradation and poor long-term stability. Moreover, manganese ions migrating to the anode surface can disrupt SEI stability via multiple mechanisms: (i) electrochemical reduction to metallic Mn, which deactivates the SEI, (ii) formation of Mn-containing ionic complexes that block lithium-ion transport pathways, and (iii) a proposed hybrid mechanism involving reduction to metallic Mn followed by partial chemical oxidation—though the metallic Mn levels remain below conventional detection

thresholds. These interfacial interactions, often referred to as “electrode crosstalk,” remain a focal point of ongoing research into LIB degradation mechanisms.

1.9 Historical Development

- First proposed by Thackeray et al. (1983) as a zero-strain host for Li^+ insertion.
- Early work demonstrated its flat plateau at ~ 1.55 V vs Li/Li^+ , offering inherent safety (no Li plating).

1.9.1 Why LTO?

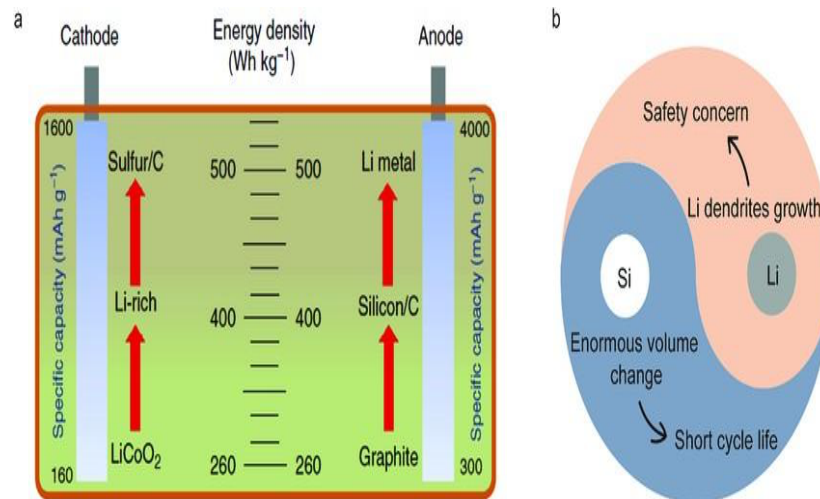


Figure 5. Comparison of Specific Capacities and Limitations of Battery Electrode Materials

- **Zero-Strain Material:** LTO is considered a "zero-strain" material because it undergoes minimal volumetric expansion ($<1\%$) during lithium intercalation and deintercalation [2]. This minimizes mechanical stress, reducing the risk of structural degradation over cycling.

- **Thermal Stability:** LTO operates safely under high temperatures and has a low risk of thermal runaway, making it a safer alternative compared to graphite anodes.
- **Fast Lithium Diffusion:** LTO has a three-dimensional lithium-ion diffusion pathway, allowing rapid lithium intercalation and deintercalation. This results in excellent high-rate performance and fast charging capability.

1.9.2 Crystal Structure and Phase Stability of LTO

Cubic Spinel Framework (Fd-3m)

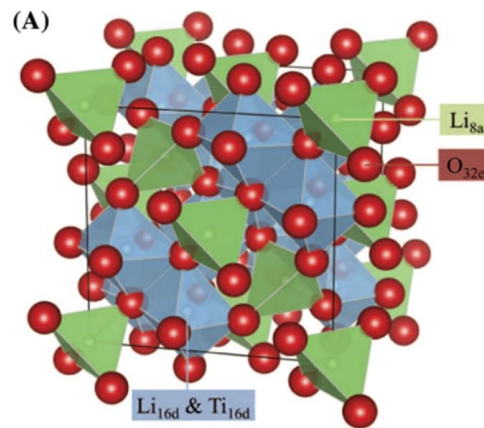


Fig. 6 Structure of LTO

- Oxygen forms a close-packed face-centered cubic lattice; Li occupies tetrahedral 8a sites, Ti occupies octahedral 16d sites.
- Upon lithiation to $\text{Li}_7\text{Ti}_5\text{O}_{12}$, additional Li populates octahedral 16c sites with minimal lattice expansion.

CHAPTER 2

Theoretical Background

2.1 Complex Impedance Spectroscopy

Complex Impedance Spectroscopy (CIS) is a versatile and non-invasive analytical method employed to explore the electrical characteristics of materials across a wide frequency spectrum. The technique operates by applying a small-amplitude AC voltage to the sample and measuring the resulting current, allowing the calculation of complex impedance, $Z(\omega)$, which consists of both resistive (real) and reactive (imaginary) components. These components provide insights into energy dissipation and storage mechanisms within the system. CIS is particularly useful for analyzing different conduction pathways—such as bulk (grain), grain boundary, and interfacial effects—and is widely utilized in the study of ceramics, dielectrics, solid-state electrolytes, and energy storage materials. It facilitates the extraction of key electrical properties like AC conductivity, dielectric permittivity, relaxation behavior, and activation energy. Impedance data are often interpreted through Nyquist or Bode plots and fitted to equivalent electrical circuit models that represent the material's underlying physical and electrochemical processes. [4]

2.2 Dielectric Relaxation Mechanisms

Dielectric relaxation describes how a dielectric material responds to an alternating electric field, particularly how polarization within the material relaxes back to equilibrium after the field is removed. The main mechanisms include:

- Electronic polarization: Displacement of electron clouds relative to nuclei, dominant at high frequencies.
- Atomic polarization: Displacement of atomic nuclei within molecules.
- Dipole relaxation: Alignment and relaxation of permanent or induced dipoles,

heavily influenced by temperature and local environment.

- Ionic relaxation: Movement of ions, including interfacial polarization (Maxwell-Wagner-Sillars effect), where charge accumulates at interfaces within heterogeneous materials.[5]

These mechanisms each have characteristic frequencies and contribute to the overall dielectric response. Dielectric relaxation is typically observed in the frequency range $10^2 - 10^{10}$ Hz [6], [7], [8], [9], [10], [11], [12], [13], [14], [15], [16], [17], [18], [19].

2.3 Rietveld Refinement Principles

Rietveld refinement is a computational method used to extract detailed structural information from powder X-ray or neutron diffraction data. The method involves fitting a calculated diffraction pattern, based on a structural model, to the observed data by minimizing the difference (using a least-squares approach) between the measured and calculated intensities across the entire pattern [8]

Key aspects include:

- Use of all data points, not just peak positions and intensities.

- Iterative refinement of parameters such as lattice constants, atomic positions, thermal factors, and instrumental parameters.
- Ability to handle overlapping reflections, making it especially valuable for complex or low-symmetry structures [8].

2.2 Key Parameters: Lattice Constants, Strain, Crystallite Size

- Lattice constants define the unit cell dimensions of a crystal and are fundamental to identifying phases and structural changes.
- Strain in the lattice can arise from defects, size effects, or external stresses, and it influences peak broadening in diffraction patterns.
- Crystallite size refers to the size of coherent, diffracting domains and can be estimated from peak broadening (e.g., using the Scherrer equation).

Notably, an increase in crystallite size generally leads to a decrease in lattice parameters, while lattice strain tends to increase with increasing crystallite size [9].

CHAPTER 3

MATERIALS AND METHOD

3.1 METHODOLOGY:

The anode compositions are synthesized by a solid-state reaction method from stoichiometric amounts of Li_2CO_3 , TiO_2 as:



The amount that was taken for Li_2CO_3 was 0.965g and 2.6205g for TiO_2 .

A slight excess amount of lithium was used to compensate for any loss of the metal which might have occurred during the calcination process. The solid-state reaction synthesis method involves one step. The precursors, as raw materials, are well mixed and thoroughly ground with agate mortar for 8 hours, then subjected to heat treatment and calcined at three different temperatures for 12 hrs. (The three different temperatures were 700°C, 800°C and 900°C) to dry the samples and sintered the pellet at 900°C for 16 h to complete the chemical reaction with air using a muffle box furnace.

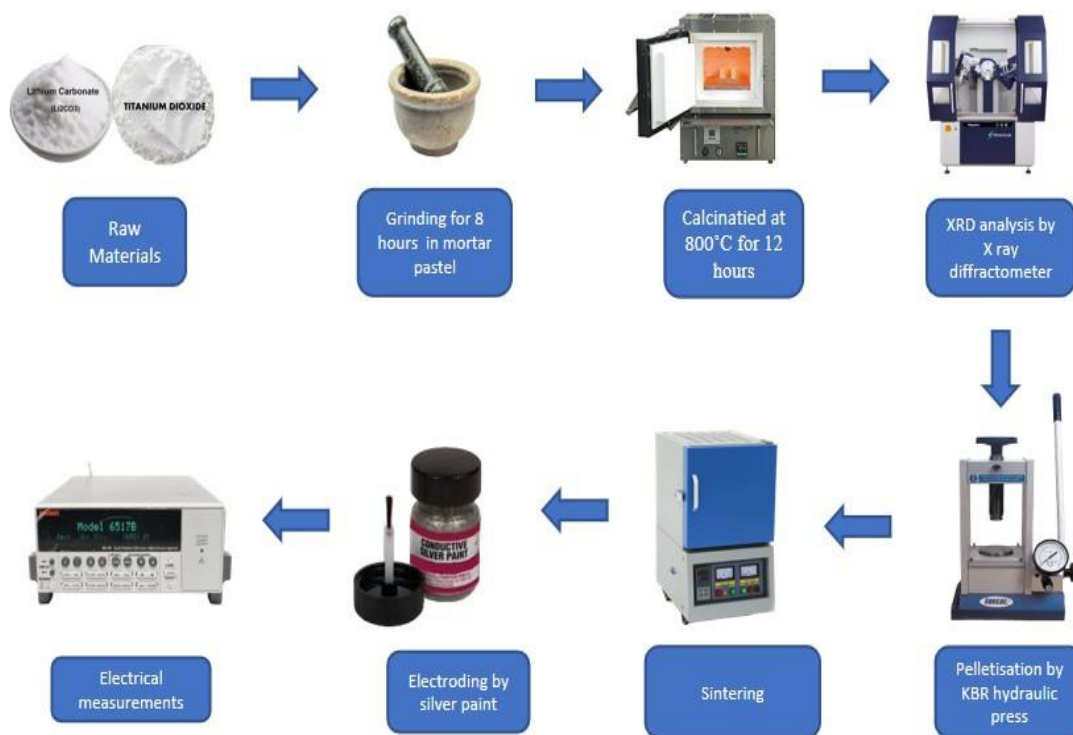


Fig. 7 Methodology of synthesis of LTO

3.1.1 MATERIAL CHARACTERIZATION:

After completing the calcination process, the sample was analyzed using X-ray diffraction to examine the crystal structure and characteristics. The analysis was conducted at 45 kV and 40 mA with Cu K α radiation ($K\alpha = 1.5456 \text{ \AA}$), employing a scanning rate of 22° per minute over a 2θ range from 10° to 90° . The data was analyzed using origin and Xpert High score for characterization of material to determine what type of crystal structure is formed. [20]

3.1.2 LCR

Impedance measurements were conducted using a computer-controlled LCR meter with an AC signal amplitude of 1.3 V. The temperature was gradually increased from 40°C

to 140 °C during the tests. Measurements were performed over a frequency range of 4 Hz to 8×10^6 Hz, with temperatures varying from 40 °C to 140 °C. Prior to testing, the leads were thoroughly examined to ensure that no unintended inductive or capacitive effects interfered with the results. An AC signal with an amplitude of 0.1 V was applied consistently throughout the experiments. Additionally, both surfaces of the pellet were thoroughly cleaned and to establish a reliable electrical contact's conductive silver paint was applied on both the opposite faces [21]. Impedance spectroscopy enables the separation of electrical parameters into their real and imaginary components, thereby facilitating a deeper understanding of the material's electrical properties. In this study, we report the complex impedance, denoted as $Z = Z' + jZ''$, here Z' and Z'' correspond to the real and imaginary components of the impedance and the graph between these two are known as Nyquist plot, respectively [22]

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 X-RAY DIFFRACTION

Figure 8 presents the X-ray diffraction (XRD) patterns for synthesized LTO samples that were calcined at temperatures ranging from 700°C to 900°C. The diffraction patterns reveal peaks that align with the standard data for $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (JCPDS card #49-0207), thereby confirming successful synthesis at 800°C and 900°C [23]. All observed peaks can be attributed to the spinel structure of LTO with an Fd-3m space group. The XRD patterns reveal that for the pure LTO sample, the intensity of the peaks is noticeably reduced when the sample is calcined at 900°C for 12 hours, and several peaks are absent in both patterns. This behavior is attributed to the formation of the impurity phase, rutile TiO_2 , which interferes with the expected diffraction peaks of LTO [24]. Available diffraction peaks with a $2\theta(\text{deg.})$ value around 18.3, 35.63, 43.23, 57.19, 62.88, 66.04, 74.39, 79.59 and 82.35 are assigned to the (1 1 1), (3 1 1), (4 0 0), (3 3 1), (3 3 3), (4 4 0), (5 3 1), (5 3 3), (6 2 2), (4 4 4), (5 5 1) and (7 3 1) planes of spinel LTO, respectively [4]. The crystalline size (D) of LTO was determined from the Bragg peaks using Scherrer's formula: $[D = \frac{K\lambda}{\beta \cos\theta}]$

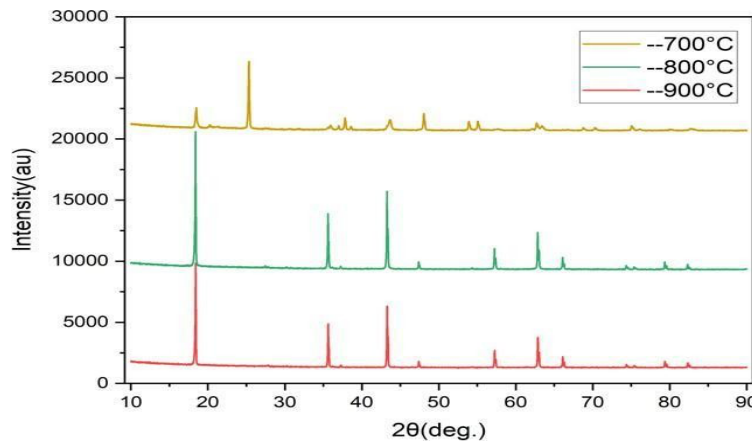


Figure 8. X-Ray Diffraction of synthesized LTO at different temperatures.

4.2 REFINEMENT

The refinement yielded a Goodness of Fit (GOF) value of 2.21, a profile R-factor (R_p) OF 4.35%, and a weighted profile R-factor (R_{wp}) OF 6.26 %. The Bragg reflections match the cubic spinel structure with space group $Fd-3m$. The Refined lattice parameters was found to be 8.3573 Å consistent with the standard values reported for cubic $\text{Li}_4\text{Ti}_5\text{O}_{12}$ [25]. The values of lattice constant confirms that the structure crystallizes in cubic system. Rietveld refinement of XRD data yielded a crystalline size of 36.3 nm and a microstrain value of -1.21 indicating the small compressive lattice distortion within the spinel framework [26]. The quality of refinement is in the acceptable limits for robust structural modeling given that (R_{wp} =6.26%, R_p =4.35%) and a goodness of fit (GOF) of 1.39. the obtained crystalline size is notably smaller than the typical values obtained for solid-state synthesized LTO (often 100-200 nm) which suggest that the synthesis conditions have limited the grain growth significantly. The observed negative microstrain is caused due to lattice defects or minor non-stoichiometry phenomena associated with high-temperature solid state synthesis which effects the electrochemical properties of LTO anodes.

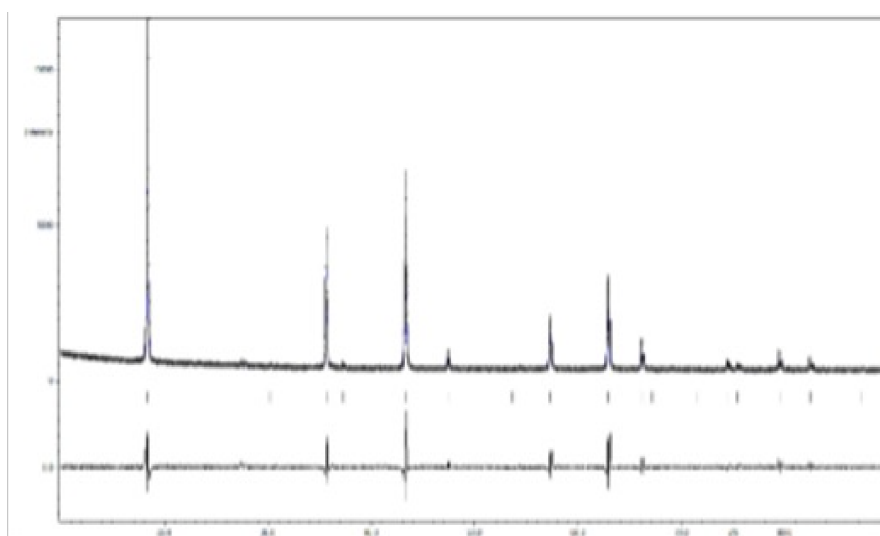


Figure 9 Diffraction profile plot of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ made using JANA 2006 software.

Refined Lattice Parameters

Table 1. listed values for cell parameters and Reciprocal Parameters

Cell Parameter	a	b	c	α	β	γ	volume
	8.3573	8.3573	8.3573	90	90	90	583.7
Reciprocal Parameters	a^{-1}	b^{-1}	c^{-1}	α	β	γ	Volume
	0.119656	0.119656	0.119656	90	90	90	0.001713

Table 2. listed values for lattice parameters and additional values after Rietveld refinement

Parameter	Value	$D = K \lambda / C$ (in $\text{\AA}^\circ$)	Microstrain
Crystal System	Cubic	362.9685864	-1.21
Cell parameter	8.3573 $\text{\AA}^0$		
Volume	583.7 $\text{\AA}^0$		
GOF	1.39		
Rp/Rwp	4.35/6.26		

4.3 Electrical properties

4.3.1 Impedance spectrum analysis

Figure 3 displays the Nyquist plot, which represents the typical impedance diagram by plotting the real part (Z') against the imaginary part (Z'') for the synthesized $\text{Li}_4\text{Ti}_5\text{O}_{12}$ sample at different temperatures with increasing frequency. The CIS enables the differentiation between grain (bulk) and grain boundary resistances because each exhibits a unique relaxation time, resulting in distinct semicircular arcs in the Nyquist plot. The spectrum reveals two semicircular arcs with centers located below the real axis. The high-frequency semicircle is predominantly linked to the bulk properties and results from the parallel combination of the material's intrinsic bulk resistance and bulk capacitance. In contrast, the low-frequency semicircular arc—highlighted in the inset of Fig. 3- is attributed to the grain boundaries, which typically exhibit higher resistance and slower charge transport characteristics. Both the grain interiors and grain boundaries exhibit unique resistance values that can be quantified by fitting the semicircular arcs in the impedance spectrum. The influence of temperature was investigated at different temperature between 40°C to 140°C. Here we show the spectra measured at four points at temperatures 140°C, 120°C, 100°C and 60°C. Fig. 4

It displays the loss spectrum, illustrating how the imaginary component of the impedance (Z'') varies with frequency at selected temperatures. This spectrum offers insights into the process characterized by the highest resistance. Distinct peaks emerge at a characteristic frequency, which varies with temperature and reflects the type and intensity of the electrical relaxation phenomenon occurring within the material. As the temperature rises, the peak shifts toward higher frequencies, likely indicating enhanced relaxation effects. Additionally, the reduction in the maximum Z'' values with increasing temperature suggests a growing loss in the material's resistive properties. This behavior is potentially attributable to the effects of space charge within the sample.[9]

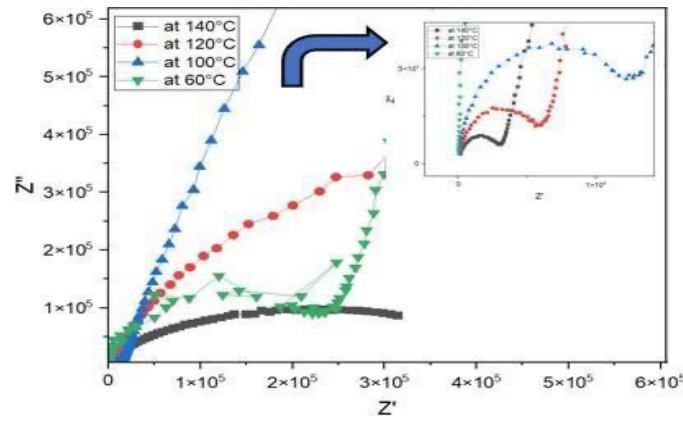


Figure 10. Complex Impedance spectrum of synthesized LTO

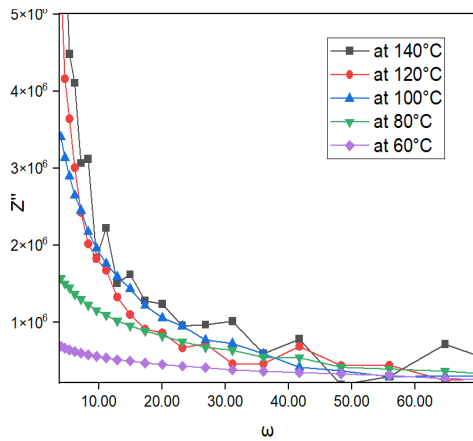


Figure 11. Frequency Dependence of Imaginary part of Impedance/loss (Z'')

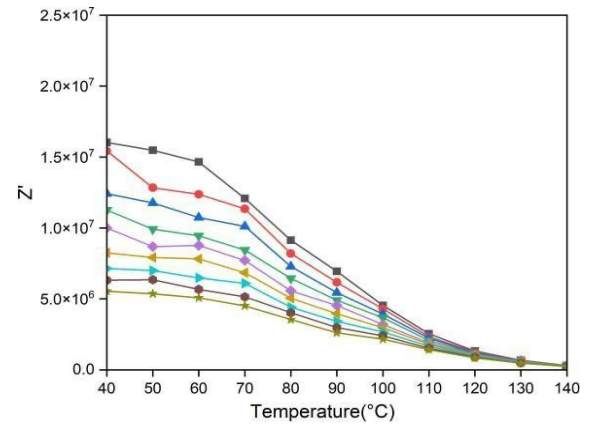


Figure 12. Temperature Dependence of Real part of Impedance/loss (Z')

Dielectric Analysis

Figure 11 shows how the dielectric constant depends on the frequency at different temperatures for LTO. Within a fixed temperature range, the dielectric constant gradually declines as the frequency increases. In contrast, raising the temperature leads to a noticeable enhancement in the dielectric constant effect that is particularly prominent at lower frequencies. This shows that the system's dielectric relaxation process is driven by thermal activation, meaning that temperature changes significantly influence its behavior. The dielectric behavior of sample of frequency is closely linked

electric field. Since an alternating electric field continually re- verses its direction, the polarization components must adapt to each reversal. Therefore, the resulting polarization is contingent upon the dipoles' ability to reorient themselves in response to every change in the field direction. At lower frequencies, the dipoles have enough time to fully realign with the direction of the applied electric field, which leads to a higher dielectric constant. However, as the frequency increases, the rapid alternation of the field means that the dipoles cannot reorient quickly enough. This lag in response causes the dielectric constant to decrease and eventually level off.

The variation of dielectric loss as a function of temperature at a frequency of 20 kHz is represented in fig 12. The dielectric loss pattern typically exhibits two distinct features. First, there is a progressive increase in loss with rising temperature, which then suddenly accelerates at a specific temperature. Second, the overall shape of the loss profile is asymmetric. The emergence of a peak in a dielectric loss versus temperature graph signifies a transition between processes at a well-defined temperature. This peak may be attributed to thermal mechanisms within the material that result in the highest possible energy dissipation per cycle relative to the stored energy. The study reveals that grain boundary resistance declines as temperature increases, which in turn enhances the dc conductivity. This behavior is plausible because the intrinsic properties of LTO appear to evolve with rising temperature, possibly leading to a reduction in polarization. This result offers valuable insight, linking the increase in conductivity with rising temperature to the effects associated with the grain boundaries. Comprehensive analysis using CIS measurements has yielded unequivocal evidence that grain-boundary phenomena predominantly govern the electrical behavior of LTO. The impedance spectroscopy results unambiguously indicate that these microstructural features exert a decisive influence on the overall electrical conduction within the material. Although our investigation has been primarily centered on the conduction aspect, the data clearly demonstrates that the grain-boundary effects are integral to the electrical properties of LTO. This insight not

only reinforces the critical role of grain boundaries in modulating the conduction mechanism but also provides a robust framework for further optimizing. This result offers valuable insight, linking the increase in conductivity with rising temperature to the effects associated with the grain boundaries. Comprehensive analysis using CIS measurements has yielded unequivocal evidence that grain-boundary phenomena predominantly govern the electrical behavior of LTO. The impedance spectroscopy results unambiguously indicate that these microstructural features exert a decisive influence on the overall electrical conduction within the material. The data clearly demonstrates that the grain-boundary effects are integral to the electrical properties of LTO. This insight not only reinforces the critical role of grain boundaries in modulating the conduction mechanism but also provides a robust framework for further optimizing the performance of LTO-based materials in various applications.[4], [9]

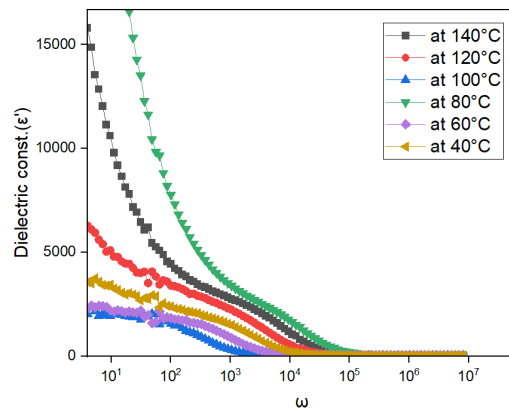


Figure13. Dielectric const. as a function of frequency at diff. temperature

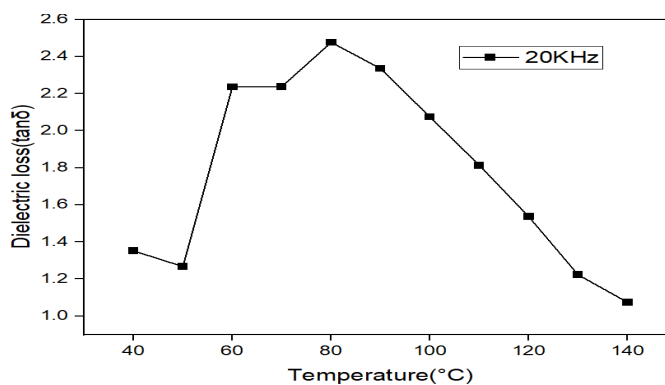


Figure14. Dielectric loss factor as a function of temperature

AC Conductivity analysis and Activation energy

The Arrhenius plot given in figure 15 shows the relation between $\log(\sigma T)$ versus $1000/T$. This relation is significant to find the thermal activation energy of charge transport in electrode materials. The increase in conductivity from 5.56×10^{-6} S/cm at 313.15 K to 6.92×10^{-5} S/cm at 393.15 K indicates the thermally active process suggesting the Li^+ migration within the LTO material is facilitated by higher thermal energy. The figure x represents the straight line observed in the Arrhenius plot. This relation shows that charge transport mechanism follows the following relation:

$$\sigma = \sigma_0 \left(e^{-\frac{E_a}{K_B T}} \right) \quad (2)$$

where σ_0 is the conductivity, E_a is the activation energy for conduction and K_B is the Boltzmann constant and T is the absolute temperature in kelvin. The activation energy is extracted from the relation to understand the conduction pathways of LTO [9]. The result shows that ionic and electronic diffusion improves at elevated temperature thereby enhancing overall conductivity. The average AC conductivity of the Sample was found to be approximately 2.6×10^{-5} S/cm and the AC conductivity was found to be 0.0122 eV for the LTO material.[4]

Table 3 The Activation Energy with temperature

Temperature	Z	Sigma	Log (σT)	1000/t
313.15	8573983.5	5.53×10^{-6}	-2.759	3.19
333.15	5040102.2	9.47×10^{-6}	-2.501	3
353.15	5481478	8.73×10^{-6}	-2.511	2.83
373.15	3407114.5	1.40×10^{-5}	-2.282	2.68
393.15	691523.44	6.92×10^{-5}	-1.566	2.54

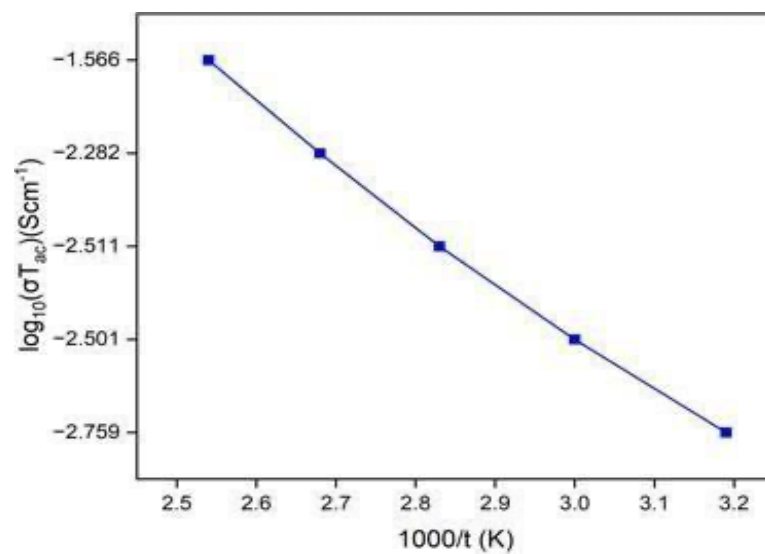


Figure 15. Arrhenius plot of $\log (\sigma T)$ vs $1000/t$ for LTO

CONCLUSION

In this work, spinel-phase $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (LTO) was successfully synthesized via a conventional solid-state method. Structural analysis using X-ray diffraction and Rietveld refinement confirmed the cubic spinel phase (Fd-3m) with high crystallinity, a lattice constant of $8.3573\text{\AA}$, and crystallite size of 36.3 nm. The material showed excellent phase purity with minimal strain, as reflected in the quality of the refinement parameters.

The impedance spectroscopy analysis revealed clear contributions from both bulk and grain boundary effects, with grain boundaries significantly influencing the overall conduction process. The AC conductivity followed Arrhenius behavior, with a calculated activation energy of 0.0122 eV, validating a thermally activated ionic transport mechanism.

Dielectric analysis showed a strong frequency and temperature dependence of permittivity and loss, governed primarily by space charge and grain boundary effects. These electrical characteristics affirm the material's suitability for high-rate lithium-ion battery anode applications, offering enhanced safety, excellent cycle life, and rapid charge-discharge capability.

Overall, this investigation provides deep insight into the interplay between structure and electrochemical behavior of LTO, establishing a solid foundation for further material optimization and battery performance enhancement.

9.2 Scientific Contributions

This work contributes to the scientific understanding of LTO by elucidating the relationships between its crystal structure, microstructural features, and electrochemical/dielectric properties. The use of advanced characterization techniques, including Rietveld refinement, impedance spectroscopy, and dielectric

analysis, provides a comprehensive view of how structural integrity and phase purity influence ionic conductivity and charge storage mechanisms. The findings highlight the importance of tailoring synthesis and post-treatment conditions to optimize crystallite size, lattice strain, and grain boundary composition. Furthermore, the study demonstrates that composite strategies and surface modifications can significantly improve electrochemical performance, offering new insights for the design of next-generation anode materials.

9.3 Practical Recommendations

Due to their excellent rate capability, structural stability, and enhanced safety profile, LTO-based anodes are particularly well-suited for high-power and fast-charging lithium-ion battery systems. These characteristics make them ideal candidates for demanding applications such as electric vehicles (EVs), hybrid electric vehicles (HEVs), and stationary energy storage systems (ESS), where rapid charge–discharge cycles and long cycle life are critical performance requirements, where safety and cycle life are critical. To address the limitations of LTO, future efforts should focus on:

Surface engineering and coating strategies (e.g., Al_2O_3 atomic layer deposition) to suppress gas evolution and improve thermal stability, particularly at elevated operating temperatures.

Development of composite materials (e.g., LTO/NiO, LTO/carbon) to enhance specific capacity and rate capability.

Optimization of electrode architecture to minimize interfacial resistance and maximize ionic/electronic conductivity.

Advanced characterization techniques (e.g., in situ XRD, TEM) to monitor structural evolution and degradation mechanisms during cycling.

Long-term cycling and safety testing under realistic conditions to ensure reliability and commercial viability.

These recommendations are expected to guide further research and industrial development of LTO-based anode materials for advanced lithium-ion batteries.

REFERENCES:

- [1] C. K. Chan *et al.*, “High-performance lithium battery anodes using silicon nanowires,” *Nat Nanotechnol*, vol. 3, pp. 31–35, Jan. 2008, doi: 10.1038/nnano.2007.411.
- [2] “Featured properties of Li⁺ -based battery anode: Li₄Ti₅O₁₂,” https://pubs.rsc.org/en/content/articlepdf/2020/ra/d0ra00818d?utm_source=chatgpt.com.
- [3] Z. Deng, Z. Xu, W. Deng, and X. Wang, “Ultrafine Li₄Ti₅O₁₂ nanocrystals as building blocks for ultrahigh-power lithium-ion battery anodes,” *J Power Sources*, vol. 521, Feb. 2022, doi: 10.1016/j.jpowsour.2021.230970.
- [4] E. Barsoukov and J. R. Macdonald, *Impedance Spectroscopy: Theory, Experiment, and Applications*. John Wiley and Sons, 2005. doi: 10.1002/0471716243.
- [5] R. A. Pethrick, “Dielectric Relaxation,” in *Techniques and Applications of Fast Reactions in Solution*, Springer Netherlands, 1979, pp. 103–113. doi: 10.1007/978-94-009-9490-4_15.
- [6] “UNIT-III-Dielectric and Magnetic properties of materials.”
- [7] “1380c8b1-8b75-4442-86b6-0ae6585e1ead”.
- [8] N. Sahu and S. Panigrahi, “Mathematical aspects of Rietveld refinement and crystal structure studies on PbTiO₃ ceramics,” 2011.
- [9] S. L., T. N., S. G., and A. P., “STUDY OF STRUCTURE AND ELECTRONIC PROPERTY OF SPINEL Li₄Ti₅O₁₂ ANODE MATERIAL,” Buryat State University, 2016, pp. 104–110. doi: 10.18101/978-5-9793-0898-2-104-110.
- [10] A. Farmann, W. Waag, and D. U. Sauer, “Application-specific electrical characterization of high power batteries with lithium titanate anodes for electric vehicles,” *Energy*, vol. 112, pp. 294–306, Oct. 2016, doi: 10.1016/j.energy.2016.06.088.
- [11] S. Dutta, R. N. P. Choudhary, P. K. Sinha, and A. K. Thakur, “Microstructural studies of (PbLa)(ZrTi)O₃ ceramics using complex impedance spectroscopy,” *J Appl Phys*, vol. 96, no. 3, pp. 1607–1613, Aug. 2004, doi: 10.1063/1.1765869.
- [12] S. L., T. N., S. G., and A. P., “STUDY OF STRUCTURE AND ELECTRONIC PROPERTY OF SPINEL Li₄Ti₅O₁₂ ANODE MATERIAL,” Buryat State University, 2016, pp. 104–110. doi: 10.18101/978-5-9793-0898-2-104-110.
- [13] F. KILIÇ DOKAN and Ş. PATAT, “The Effect of Synthesis Parameters on the Electrochemical Performance of Li₄Ti₅O₁₂,” *Erzincan Üniversitesi Fen Bilimleri Enstitüsü Dergisi*, vol. 15, no. 2, pp. 482–497, Aug. 2022, doi: 10.18185/erzifbed.979824.
- [14] G. Q. Liu *et al.*, “Synthesis and electrochemical properties of Li₄Ti₅O₁₂.”
- [15] B. Vikram Babu *et al.*, “Structural and electrical properties of Li₄Ti₅O₁₂ anode material for lithium-ion batteries,” *Results Phys*, vol. 9, pp. 284–289, Jun. 2018, doi: 10.1016/j.rinp.2018.02.050.
- [16] N. V Prasad, G. Prasad, T. Bhimasankaram, S. V Suryanarayana, and G. S. Kumar, “Synthesis, impedance and dielectric properties of LaBi₅Fe₂Ti₃O₁₈,” 2001.
- [17] S. Dutta, R. N. P. Choudhary, P. K. Sinha, and A. K. Thakur, “Microstructural studies of (PbLa)(ZrTi)O₃ ceramics using complex impedance spectroscopy,” *J Appl Phys*, vol. 96, no. 3, pp. 1607–1613, Aug. 2004, doi: 10.1063/1.1765869.
- [18] V. Natalia, A. P. Gustami, F. Rahmawati, W. W. Lestari, and A. Purwanto, “Lithium titanate (LTO) synthesis through solid state reaction and its performance for LiFePO₄/LTO battery,” *Journal of Mathematical and Fundamental Sciences*, vol. 50, no. 3, pp. 290–302, 2018, doi: 10.5614/j.math.fund.sci.2018.50.3.5.
- [19] R. Acevedo, “Basics of Impedance Spectroscopy,” 2019.
- [20] S. Garcia-Granda and J. M. Montejo-Bernardo, “X-Ray Diffraction - Powder,” in *Encyclopedia of Analytical Science: Second Edition*, Elsevier Inc., 2004, pp. 389–397. doi: 10.1016/B0-12-369397-7/00671-3.
- [21] H. Dalshad Omar, “To Investigation the Structure and Morphology of Iron Metallic by Difference Techniques,” *J. Nano. Adv. Mat*, vol. 3, no. 2, p. 57, 2015, doi: 10.12785/jnam/030201.
- [22] J. R. Macdonald, “Impedance Spectroscopy,” 1992.
- [23] B. Vikram Babu *et al.*, “Structural and electrical properties of Li₄Ti₅O₁₂ anode material for lithium-ion batteries,” *Results Phys*, vol. 9, pp. 284–289, Jun. 2018, doi: 10.1016/j.rinp.2018.02.050.
- [24] N. Zhang, Z. Liu, T. Yang, C. Liao, Z. Wang, and K. Sun, “Facile preparation of nanocrystalline Li₄Ti₅O₁₂ and its high electrochemical performance as anode material for lithium-ion batteries,” *Electrochem commun*, vol. 13, pp. 654–656, Jun. 2011, doi: 10.1016/j.elecom.2011.03.038.
- [25] X. Bai, W. Li, A. Wei, Q. Chang, L. Zhang, and Z. Liu, “Preparation and electrochemical performance of F-doped Li₄Ti₅O₁₂ for use in the lithium-ion batteries,” *Solid State Ion*, vol. 324, pp. 13–19, Oct. 2018, doi: 10.1016/j.ssi.2018.06.005.
- [26] S. Murugesan, R. Thirumurugesan, E. Mohandas, and P. Parameswaran, “X-ray diffraction Rietveld analysis and Bond Valence analysis of nano titania containing oxygen vacancies synthesized via sol-gel route,” *Mater Chem Phys*, vol. 225, pp. 320–330, Mar. 2019, doi: 10.1016/j.matchemphys.2018.12.061.

APPENDICES

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REFERENCES

- Thackeray, M. M., David, W. I. F., Bruce, P. G., & Goodenough, J. B. (1983). Lithium insertion into spinel oxides. *Materials Research Bulletin*, 18(4), 461–472.
- Ohzuku, T., Ueda, A., & Nagayama, M. (1995). Electrochemistry and structural chemistry of Li insertion into $\text{Li}_4\text{Ti}_5\text{O}_{12}$. *Journal of the Electrochemical Society*, 142(5), 1431–1435.
- Kavan, L., Grätzel, M., Gilbert, S. E., Klemenz, C., & Scheel, H. J. (1996). Electrochemical and photoelectrochemical properties of TiO_2 (anatase). *Journal of the American Chemical Society*, 118(28), 6716–6723.

- Julien, C. M., Mauger, A., Zaghib, K., & Groult, H. (2014). Comparative issues of cathode materials for Li-ion batteries. *Inorganics*, 2(1), 132–154.
- Liu, J., et al. (2011). A review of recent developments in Li-ion battery cathode materials. *Journal of Power Sources*, 196(18), 7394–7403.
- Sun, Y. K., et al. (2002). Preparation and electrochemical behavior of nano-crystalline $\text{Li}_4\text{Ti}_5\text{O}_{12}$. *Electrochemistry Communications*, 4(6), 344–347.
- Li, W., Reimers, J. N., & Dahn, J. R. (1993). Solid electrolyte interphase on graphite anodes in Li-ion batteries. *Solid State Ionics*, 67(1), 123–130.
- Wang, Y., et al. (2008). Nanostructured $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as anode materials for high power lithium ion batteries. *Journal of Power Sources*, 184(2), 654–659.
- Tang, Y., Zhang, Y., Li, W., & Song, D. (2009). Nanostructured $\text{Li}_4\text{Ti}_5\text{O}_{12}$: synthesis and lithium storage properties. *Journal of Power Sources*, 189(2), 758–761.
- Zhang, X., et al. (2011). Nano- $\text{Li}_4\text{Ti}_5\text{O}_{12}$ synthesized by sol–gel method and its performance in Li-ion batteries. *Journal of Alloys and Compounds*, 509(5), 2560–2565.
- Yang, X., et al. (2013). Carbon-coated $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode for high performance Li-ion batteries. *Electrochimica Acta*, 111, 346–353.
- Bresser, D., Passerini, S., & Scrosati, B. (2013). Lithium titanate ($\text{Li}_4\text{Ti}_5\text{O}_{12}$) anode material for lithium-ion batteries. *Chemical Communications*, 49(90), 10545–10562.
- Tarascon, J. M., & Armand, M. (2001). Issues and challenges facing rechargeable lithium batteries. *Nature*, 414(6861), 359–367.
- Liu, H., Wang, G., Liu, J., & Qiao, S. (2014). Electrochemical performance of LTO anode modified by surface carbon. *Journal of Materials Chemistry A*, 2(9), 3101–3109.
- Song, J., & Cairns, E. J. (2012). Thermal and electrochemical stability of LTO in lithium-ion cells. *Journal of the Electrochemical Society*, 159(7), A1032–A1037.
- Aravindan, V., Gnanaraj, J., Lee, Y. S., & Madhavi, S. (2011). Insertion-type electrodes for nonaqueous Li-ion capacitors. *Chemical Reviews*, 114(23), 11619–11635.
- Ouyang, C. Y., Song, L. J., Wang, Z. X., & Huang, X. J. (2007). First-principles study of Li-ion diffusion in $\text{Li}_4\text{Ti}_5\text{O}_{12}$. *Electrochemistry Communications*, 9(5), 1001–1006.
- Liu, G. Q., et al. (2007). Synthesis and electrochemical properties of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. *Journal of Power Sources*, 174(2), 1075–1078.
- Yu, X., et al. (2016). Electrochemical impedance spectroscopy for LTO systems. *Electrochimica Acta*, 188, 661–669.
- Song, H. K., et al. (2003). Electrochemical performance of nano-structured spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ synthesized by a hydrothermal method. *Electrochemical and Solid-State Letters*, 6(11), A236–A239.
- Kumar, Y., et al. (2020). Structural and dielectric properties of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ prepared by solid-state reaction. *Materials Today: Proceedings*, 27, 554–559.
- Lee, Y. H., et al. (2011). Surface-modified LTO by atomic layer deposition. *Journal of Power Sources*, 196(8), 4094–4099.
- Meini, S., et al. (2014). Electrode/electrolyte interface degradation in LTO-based Li-ion batteries. *Journal of the Electrochemical Society*, 161(9), A1364–A1370.
- Huang, Y. H., Goodenough, J. B., & Park, K. S. (2008). Superior capacity of spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ electrodes. *Journal of Power Sources*, 185(2), 1209–1213.
- Yin, Y. X., Guo, Y. G., & Wan, L. J. (2009). Nanostructured Li-ion battery electrodes. *Accounts of Chemical Research*, 46(11), 2338–2348.
- Liu, H., et al. (2015). Graphene–LTO hybrid materials for advanced anode applications. *Nano Energy*, 12, 709–718.
- Lu, X., et al. (2012). Advanced LTO composites for high-rate battery performance. *Journal of Materials Chemistry*, 22(43), 23201–23207.
- Lee, J., et al. (2005). Impedance analysis of Li-ion battery using LTO anodes. *Journal of Power Sources*, 146(1–2), 198–202.
- Shaju, K. M., & Bruce, P. G. (2008). Nanostructured LTO: synthesis and performance. *Advanced Materials*, 20(5), 913–917.
- Notten, P. H., et al. (2007). 3D LTO electrode architectures for high-power applications. *Advanced Materials*, 19(24), 4564–4567.