

Synthesis and Electrochemical Properties of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as Anode Material for Lithium Ion Batteries



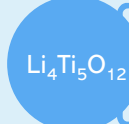
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Introduction



Lithium Ion Batteries are the most popular consumer electronic batteries, having High Energy Density and Low Self Discharge



Lithium Titanate (LTO) is anode material for Lithium Ion Batteries. It has good safety characteristic and better cycling capability.

Problems

Low Electrical Conductivity
 $\sim 10^{-13} \text{ Scm}^{-1}$

Low Theoretical Capacity
 $\sim 175 \text{ mAhg}^{-1}$

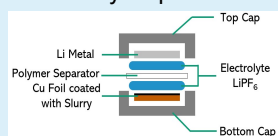
Aims

- To determine which method of synthesis is best for producing LTO batteries
- To investigate the effects of Fluorine doping on LTO

Methodology

Methods	LTO	Reactants	Conditions
Ball Milling	1	$4\text{Li}_2\text{CO}_3, 5\text{TiO}_2$	Ball Milling $780^\circ\text{C}, 6\text{h}$
	2	$4\text{LiNO}_3, 5\text{TiOSO}_4, 9.5\text{Citric acid}, 50\text{ml water}$	$800^\circ\text{C}, 3\text{h}$
Solid State	3	$4\text{LiNO}_3, 5\text{TiOSO}_4, 9.5\text{Citric acid}, 50\text{ml water}, 20\text{ml HCl}$	$800^\circ\text{C}, 3\text{h}$
	4	$4\text{Li(Acetate)}, 5\text{TiO}_2$	$800^\circ\text{C}, 3\text{h}$
Molten Salt	5	$5\text{TiO}_2, 5\text{LiCl}, 5\text{KCl}$	$800^\circ\text{C}, 6\text{h}$, Filtration
	6	$5\text{TiO}_2, 4.35\text{LiCl}, 1.15\text{KCl}, 4.5\text{LiOH}$	$800^\circ\text{C}, 6\text{h}$, Filtration
	7	$5\text{TiO}_2, 7.5\text{LiCl}, 7.5\text{KCl}$	$800^\circ\text{C}, 6\text{h}$, Filtration
	8	$5\text{TiOSO}_4, 7.5\text{LiCl}, 7.5\text{KCl}$	$800^\circ\text{C}, 6\text{h}$, Filtration
Fluorine Doping	9	2g LTO 1 + 0.4g polyvinilidene fluoride	Grinding $750^\circ\text{C}, 6\text{h}$

Battery Preparation



Scanning Electron Microscopy

X-Ray Diffraction

Galvanostatic Cycling

Cyclic Voltammetry

Impedance Spectroscopy

Electrochemical tests

Galvanostatic Cycling

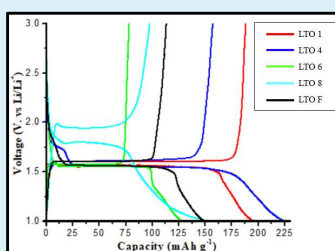


Fig 1a: Voltage against Capacity graph for LTO with good rate capability

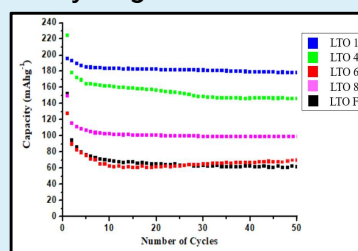


Fig 1b: The discharge capacities for 50 cycles for LTO 1, 4, 6, 8 and F

Figure 1a:

- LTO 4 showed highest initial discharge capacity (225 mAhg^{-1})
- LTO 1 (96.3%) and LTO 4 (75.8%) showed high coulombic efficiencies
- Figure 1b:
- LTO 1 and 4 showed high capacity retention (less than 20% capacity fade)

Cyclic Voltammetry

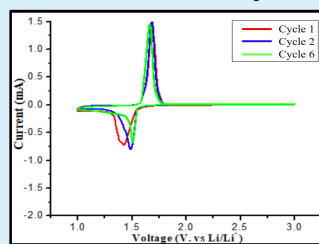


Fig 2: Typical good cyclic voltammogram

Cathodic Peak: $\sim 1.43\text{v}$
 $\text{Li}_4\text{Ti}_5\text{O}_{12} + 3\text{Li}^+ \rightarrow \text{Li}_7\text{Ti}_5\text{O}_{12}$
 Anodic Peak: $\sim 1.69\text{v}$
 $\text{Li}_7\text{Ti}_5\text{O}_{12} \rightarrow \text{Li}_4\text{Ti}_5\text{O}_{12} + 3\text{Li}^+$

LTO 1, 4, 6, F shows a good cyclic voltammograms, explaining good rate capability.

LTO 1, 4, 6 and F showed good Lithium ion diffusion quotient [Tab 1]

LTO	1	2	3	4	5	6	7	8	F
$D/\text{cm}^2 \text{ s}^{-1}$	3.6×10^{-13}	1.7×10^{-15}	2.8×10^{-15}	3.3×10^{-13}	2.3×10^{-15}	3.0×10^{-13}	8.1×10^{-16}	3.9×10^{-15}	1.4×10^{-13}

Table 1: Lithium Ion Diffusion quotient

Characterization

X Ray Diffraction

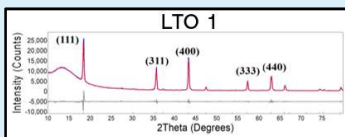
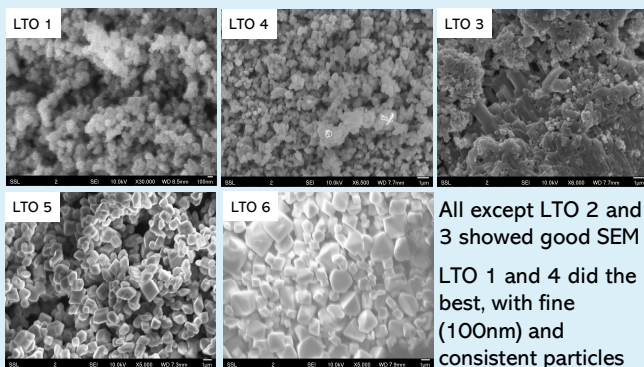


Fig 4: Good XRD of LTO (1)

Besides LTO 1, LTO 4, 6 and F showed high purity samples
 All samples showed lattice parameter close to that of 0.8355nm , indicating good purity

Scanning Electron Microscopy



All except LTO 2 and 3 showed good SEM
 LTO 1 and 4 did the best, with fine (100nm) and consistent particles

Impedance

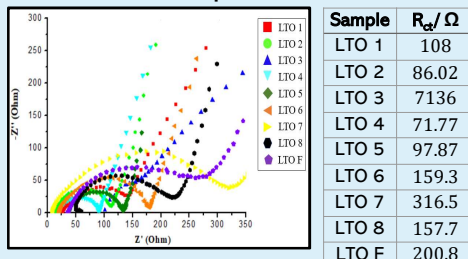


Fig 3: Nyquist plots of LTO Table 2: Rct values of LTO
 LTO 1, 2, 4, 5, 6, 8, F have good charge Transfer resistances

Sample	R_{ct}/Ω
LTO 1	108
LTO 2	86.02
LTO 3	7136
LTO 4	71.77
LTO 5	97.87
LTO 6	159.3
LTO 7	316.5
LTO 8	157.7
LTO F	200.8

Conclusion

- LTO 1, 4 and 6 performed best.
- A grinding intensive methodology can improve the electrochemical performance of LTO

Credits

I would like to thank
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 2. Organizers of IPS
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 For giving me this opportunity and supporting me.

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