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Performance of LTO/rGO Composite Anode in Lithium Ion Battery

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Abstract: $Li_4Ti_5O_{12}$ (LTO) has attracted extensive research interest as an anode material owing to its excellent safety and cycling stability. The relatively high operating potential of LTO (1.55 V vs. Li/Li^+) prevents dendrite formation, while its being above the potential at which most electrolytes and solvents are reduced leads to the formation of a thinner solid–electrolyte interphase (SEI) layer on its surface. However, the intrinsically low electronic conductivity of LTO remains the primary factor limiting its capacity. In this study, reduced graphene oxide (rGO) was synthesized and incorporated into LTO at approximately 10 wt.%, and the electrochemical performance of the resulting LTO/rGO composite anode was evaluated in lithium-ion batteries. The results revealed that rGO incorporation enhanced the discharge capacity of the LTO anode by at least 30%, while maintaining a Coulombic efficiency of nearly 100% over 100 cycles and achieving a capacity retention rate of no less than 96%.

Keywords: Lithium-Ion battery, LTO anode, rGO

Introduction

The spinel-structured intercalation-type anode, $Li_4Ti_5O_{12}$ (LTO), has been extensively investigated by researchers due to its superior safety and cycling stability compared to the graphite anode. As a result of these studies, LTO has succeeded in achieving the second largest market share in the Li-ion battery industry, following graphite (Zhang et al., 2022). The superior properties of the LTO anode primarily originate from the $Li_4Ti_5O_{12} \leftrightarrow Li_7Ti_5O_{12}$ phase transition that occurs during charge/discharge reactions (Ge et al., 2009). These two phases, belonging to the Fd3m space group, have lattice parameters of 8.3596 Å and 8.3538 Å, respectively, and the volume change during charge/discharge is less than 0.1%. This negligible volume variation imparts LTO with its remarkable cycling stability (Ariyoshi et al., 2005). In addition, the high operating potential of LTO (1.55 V vs. Li/Li^+) not only suppresses dendrite formation but also remains higher than the potential at which most electrolytes and solvents undergo reduction (< 1.0 V). This results in the formation of a thinner solid–electrolyte interphase (SEI) layer on the surface (He et al., 2012; Zhang et al., 2022). In contrast, SEI layers that typically form with greater thickness consume active material, thereby leading to capacity loss in the electrode and lower Li^+ diffusion (Zhang et al., 2022).

However, the LTO anode suffers from its extremely low electrical conductivity ($\sim 10^{-13}$ S cm^{-1}) and ionic conductivity (10^{-13}–10^{-9} cm^2 s^{-1}) (Julien & Mauger, 2024). In this study, reduced graphene oxide (rGO) was synthesized, and the electrochemical performance of the LTO/10 wt.% rGO composite anode was evaluated in lithium-ion batteries. The composite anode exhibited a substantial increase in discharge capacity without any deterioration in Coulombic efficiency or capacity retention.

- Selection and peer-review under responsibility of the Organizing Committee of the Conference

Method

Synthesis and Characterization

Graphene oxide (GO) was synthesized by mixing H_2SO_4/H_3PO_4 (360:40 mL), followed by the slow addition of $KMnO_4$ (18.0 g) and graphite flakes (3.0 g). The reaction mixture was then heated to 50 °C and stirred at 300 rpm for 12 h. After cooling to room temperature, it was poured onto ice (400 mL) with the dropwise addition of H_2O_2 (3 mL). The product was subsequently filtered and washed. The initial washing was carried out with 30% HCl solution and repeated until the supernatant became transparent, followed by further washing with deionized (DI) water and ethanol until neutral pH was achieved. All washing steps were performed by centrifugation at 6000 rpm for 30 min. The resulting solid was dispersed in DI water by ultrasonication to obtain a 1.0 mg mL^{-1} suspension. Prior to electrode preparation, the synthesized rGO (10 wt.%) was physically mixed with LTO using an agate mortar. X-ray diffraction (XRD) analyses were performed on a PANalytical Empyrean diffractometer with Cu Kα radiation at a scanning rate of 2° min^{-1}.

Electrode Preparation and Electrochemical Tests

For electrode preparation, the active material (75 wt%), Super P (15 wt%), and PVDF (10 wt%) were mixed with 0.8 mL of NMP in a centrifuge tube. The mixture was then homogenized using a vortex mixer until a slurry was obtained. The slurry was coated onto C-coated Al foil using a doctor blade with a wet thickness of 150 µm and subsequently dried at 80 °C for 24 h under vacuum-controlled atmosphere. The coated C-coated Al foil was then punched into 16 mm discs using an electrode cutting machine and further dried under the same vacuum-controlled conditions prior to cell assembly.

All the test assemblages were carried out in an Ar-filled glove box with H_2O and O_2 levels less than 0.1 ppm. Lithium metal was used as the counter electrode, while polipropilen (Celgrad 2400) membrane served as the separator. The electrolyte was 1.2 M $LiPF_6$:EC:DMC (4:6 by volume). The galvanostatic discharge and charge test cut-off potentials were 1.0 $V_{Li+/Li}$ and 3.0 $V_{Li+/Li}$, respectively. The number of cycles was set to 100 at the selected C-rates.

Results and Discussion

Structural Characterization

The XRD patterns of the freshly prepared LTO and composite electrodes on the carbon-coated Al surface are presented in Figure 1. The peaks of LTO indicates the strong crystalline structure of LTO. The incorporation of rGO does not significantly alter the XRD pattern of LTO in Figure 1, as the nanostructured nature of rGO leads to peak broadening around 2θ ≈ 26°.

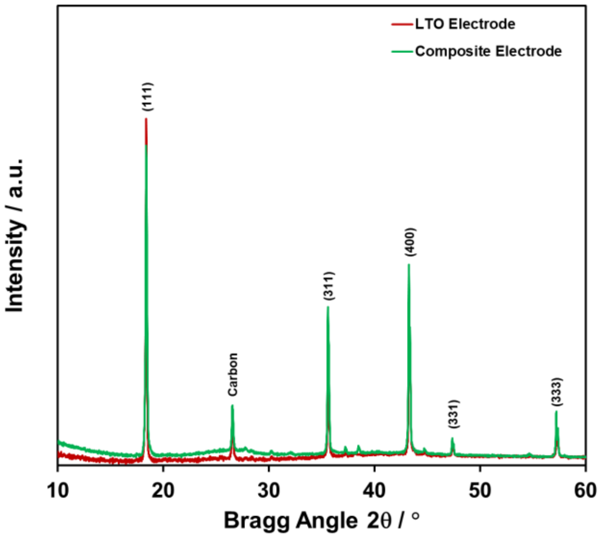

Figure 1. XRD patterns of the freshly prepared LTO and composite electrodes.

Electrochemical Tests

Figure 2 presents the effect of incorporating 10 wt.% rGO into the anode of a LiB half-cell on its charge–discharge capacities at a 1 C rate. The half-cell with a pristine LTO anode delivers a capacity of 120 mA h g^{-1}, whereas the incorporation of rGO into LTO (composite anode) increases the capacity to 156 mA h g^{-1}. This enhancement can be attributed to the layered structure and high electrical conductivity of rGO, which collectively improve the electrochemical performance of LTO.

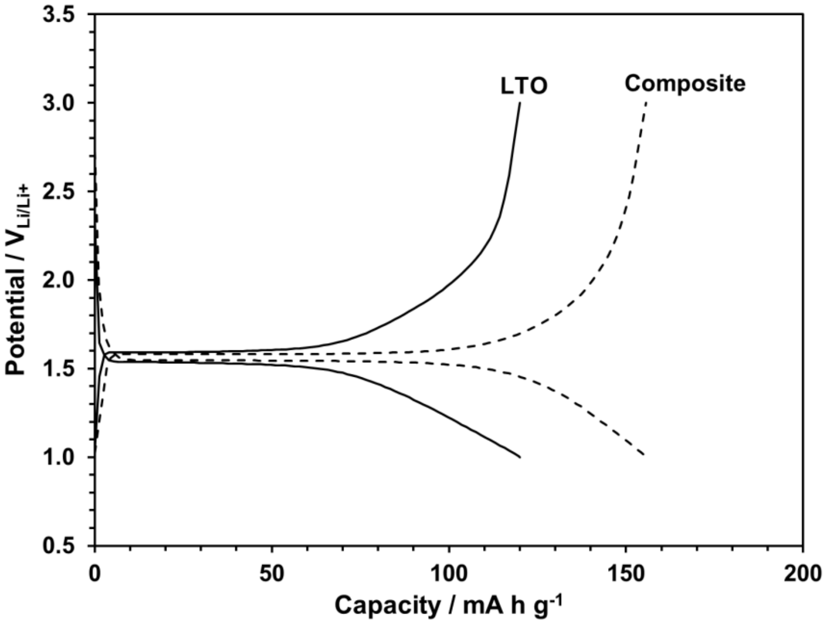

Figure 2. The charge–discharge curves of LTO and composite anode LiB half-cell at a 1 C rate.

The evolution of charge–discharge capacities during prolonged cycling is presented for the LTO and composite anodes in Figures 3a and 3b, respectively. For clarity, only the 1st, 50th, and 100th charge–discharge profiles are shown for both electrodes. The LTO anode exhibits nearly 100% Coulombic efficiency with only minimal capacity fading after 100 cycles. As depicted in Figure 4, LTO retains 96% of its initial capacity after 100 cycles. Although the composite anode demonstrates an approximately 30% increase in capacity (Figure 3b), it is also able to maintain Coulombic efficiency at 100% and a capacity retention of 97% after 100 cycles (Figure 4). These results clearly indicate that the incorporation of rGO significantly enhances the capacity of the anode without compromising Coulombic efficiency or long-term cycling stability. Figures 3 and 4 demonstrate that LTO/rGO composite electrodes are highly promising anode materials for high-performance LiBs.

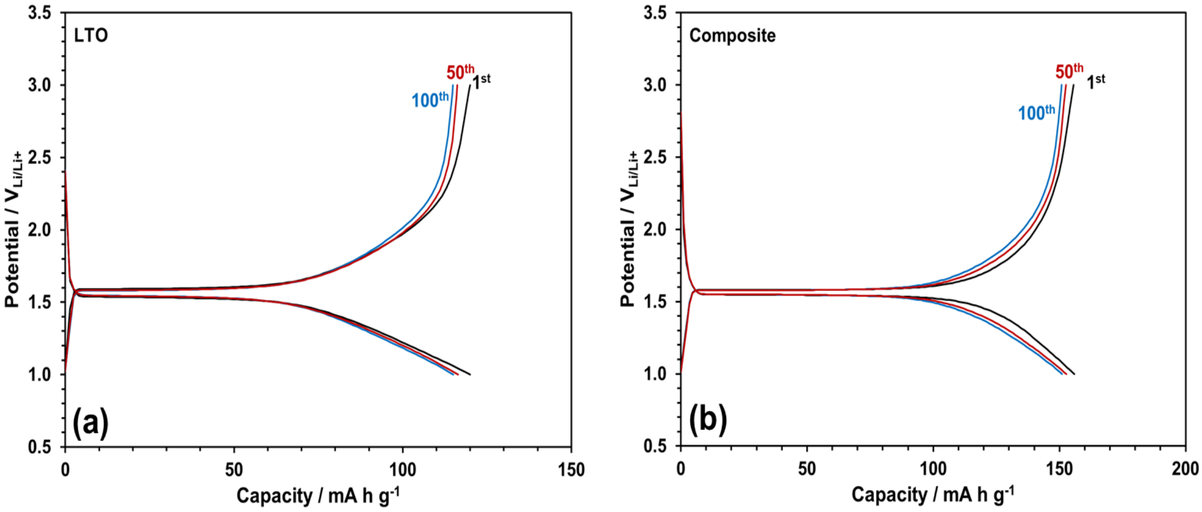

Figure 3. The 1st, 50th and 100th cycle charge–discharge curves of (a) LTO and (b) composite anode LiB half-cell at a 1 C rate.

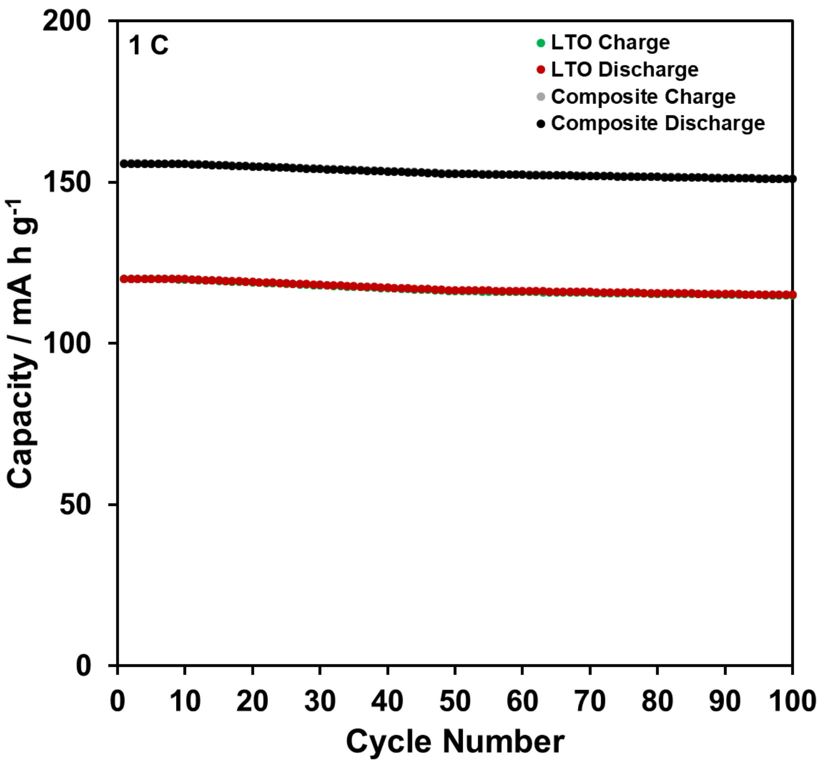

Figure 4. The change in both charge and discharge capacities over 100 cycles for LTO and composite anode LiB half-cell at a 1 C rate.

Conclusion

In this work, reduced graphene oxide (rGO) was prepared and introduced into LTO at a proportion of about 10 wt.%. The electrochemical behavior of the fabricated LTO/rGO composite anode was then examined for application in lithium-ion batteries. Findings demonstrated that the presence of rGO improved the discharge capacity of the LTO electrode by roughly 30%, while sustaining nearly 100% Coulombic efficiency across 100 charge–discharge cycles and retaining at least 96% of its initial capacity.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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