New Mixing Method to Prepare Li₄Ti₅O₁₂ Material: Structure and Electrochemical Performance

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Introduction

 $\rm Li_4Ti_5O_{12}$ has a good reversibility and experiences no structural change (zero-strain insertion material) during the charge discharge cycling. Thus $\rm Li_4Ti_5O_{12}$ is a good candidate as negative electrode for solid state lithium ion batteries and supercapacitor. In this work, we report a new synthesis method of zero-stain insertion material with carbon additive.

The present paper is an extending of our previous studies^[1, 2] on $Li_4Ti_5O_{12}$ by providing a new synthesis method. The analysis of a series of materials with different microstructures and morphologies obtained by this synthesis procedure will be presented.

Experimental

The precursors materials Ti O₂ (Anatase), Li₂CO₃ and carbon are mixed with new method and then heat treated at 850°C in flowing nitrogen to obtain the desired phases of Li₄Ti₅O₁₂. X-ray diffraction and SEM were used to analyze the products and to identify TiO₂ residue in the final powder and compared to other preparation methods. The weight loses of ternary powders was monitored by TGA to optimize the reaction temperature for synthesis. The electrochemical studies were carried out on electrodes prepared from slurries containing Li₄Ti₅O₁₂, carbon black and PVDF binder that was coated on Al Exmet. The electrodes were dried under vacuum at 85°C for 24 h before use. The studies were performed in 4-cm² laboratory cells (two electrodes) with Li counter electrodes;

Li metal(-)/EC-DMC-LiClO₄/Li₄Ti₅O₁₂(+).

The discharge-charge cycles and slow cyclic voltammetry were carried out galvanostatically and potentiostatically (Macpile^R, Claix, France). All experiments were performed at 25° C.

Results

The mixed precursor powder shows very homogonous coloration compared to the conventional mixing methods, which is an indication intimated mixing for a successful synthesis. After firing, the samples were gray colored, indicating that the samples were electronic conductors. The SEM image (Fig.1), shows dispersed carbon particles around Li-titanate particles. This carbon particles form an electronic network between the lithium titanate particles. During discharge, the voltage drops quickly to below 2 V and decreases as the reaction proceeds until the voltage reaches about 1.5 V. The cells were cycled between 1.2 V and 2 V. The results suggest that $Li_4Ti_5O_{12}$ has a very good cycle life

References

- 1 A. Guerfi, S. Sévigny and K. Zaghib , 11 IMBL,
- Monterey CA, June 2002, abstract N° 45.
- 2 A. Guerfi , S. Sévigny, M. Lagacé, P. Hovington and K. Zaghib, The 100th Meeting of the Electrochem. Soc., Philadelphia, Extended Abstract N° 823, May 2002.

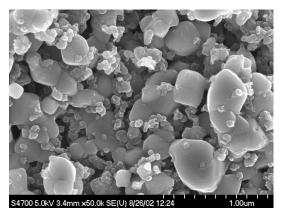


Figure1: carbon particles surrounded Li-titanate particles