Influence of Carbon on the Structure and Electrochemical Performance of Li$_4$Ti$_5$O$_12$ Electrodes

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Introduction
Li$_4$Ti$_5$O$_12$ has a good reversibility and experiences no structural change (zero-strain insertion material) during the charge discharge cycling. Thus Li$_4$Ti$_5$O$_12$ is a good candidate as negative electrode for solid state lithium ion batteries and liquid type. In this work, we report on synthesis of zero-strain insertion material with carbon additives. The influence of carbon of the structure, particle morphology and particle size were investigated. of the material and its particle shape and size are studied. The intent of the present paper is to extend our previous studies on Li$_4$Ti$_5$O$_12$ by providing a systematic analysis of a series of materials with different microstructures and morphologies obtained by a new synthesis procedure.

Experimental
The Li$_4$Ti$_5$O$_12$ (Li-Ti-O ternary phase) powders were prepared from TiO$_2$ (Anatase), Li$_2$CO$_3$ and carbon that was mixed by two methods: jar milling and ball milling. These mixtures were then heat treated at 850°C in flowing nitrogen to obtain the desired phases. X-ray diffraction and SEM were used to analyze the products and to identify TiO$_2$ residue in the final powder. 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 Li$_4$Ti$_5$O$_12$, 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-cm$^2$ laboratory cells (two electrodes) with Li metal(-)/EC-DMC-LiClO$_4$/Li$_4$Ti$_5$O$_12$(+). All experiments were performed at 25°C.

Results
The color of the samples obtained by both methods were gray, indicating that the samples were electronic conductors. The SEM images (Fig. A, B) show that different particles shapes are obtained, depending on the carbon precursor used in the synthesis of Li$_4$Ti$_5$O$_12$. The 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$_4$Ti$_5$O$_12$ has a very good cycle life. The wide variety of particle sizes (from nano to sub-micron) and shapes that were obtained leads us to conclude the synthesis procedure is suitable for optimization of the physical properties of the particles.

References

A) With carbon black (50m$^2$/g)
B) With high surface area carbon (2000m$^2$/g)