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Microstructural and Electrochemistry Study of $La_{0.50}Li_{0.50}TiO_3$

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Abstract – $La_{0.50}Li_{0.50}TiO_3$ powders has been synthesized by polymeric precursor method. The thermal evolution of the precursor powder was followed by means of Raman spectroscopy. The crystallization process of the powders heated from 350°C to 700°C was analyzed by X-ray powder diffraction (XRD). Rietveld analysis (Table 1) showed that during the crystallization process a highly crystalline perovskite phase is identified. However, a secondary phase, $LiTi_2O_4$, was also observed. The morphology of powders was observed by scanning electron microscopy - field emission gun (SEM-FEG) (Fig. 1). For the electrochemical measurements, the chronopotentiometry technique was used for testing the charge-discharge curves.

Li ion-conducting materials have been widely studied in the last few years because of their potential applications as solid electrolytes in high energy batteries and other electrochemical devices [1]. The perovskite-type lithium lanthanum titanate, $La_{2/3-x}Li_{3x}TiO_3$, is well known to be a high lithium ionic conductor having bulk conductivity of 10^{-3} Scm^{-1} at room temperature for x = 0.10. Due to this high ionic conductivity, these compounds have been studied for long time to explore the conduction mechanism and their potential use in various applications [2]. However the preparation of LLTO is generally accomplished through high temperature ceramic methods and one of the serious problems in processing LLTO by the traditional solid-state reaction route is the severe loss of lithium components due to its significant vapor pressure at the processing conditions, which makes difficult to control the stoichiometry [3]. The polymeric precursor process is an attractive method to prepare ceramic powders, offering some advantages over other methods, such as accurate stoichiometric control, good compositional homogeneity, high purity, low-temperature processing and low cost [4]. For these reasons $La_{0.50}Li_{0.50}TiO_3$ powders has been synthesized by polymeric precursor method. The effect of thermal treatment on the structure of powders has been studied by Raman spectroscopy. The Rietveld refinement of XRD patterns identified the perovskite phase $La_{0.50}Li_{0.50}TiO_3$ (P4/mmm). However, it was also observed that the crystallization of the main phase, LLTO, was accompanied by the crystallization of a secondary phase, identified as $LiTi_2O_4$. SEM-FEG analysis revealed an agglomerate morphology of homogeneous spherical particles. The electrochemistry study was performed by means a Swagelok cell configuration using a $La_{0.50}Li_{0.50}TiO_3$ pellet as cathode, a mixture of lithium perchlorate, ethylene carbonate and dimethyl carbonate as a liquid electrolyte, and lithium as anode material. Analyses of voltammetry at constant current (chronopotentiometry) were performed for testing the charge-discharge curves. The experiments revealed that the deintercalation and intercalation processes of lithium ions are not fully reversible in the $La_{0.50}Li_{0.50}TiO_3$ structure.

Table 1: Refined Crystal Parameter, Density and Volume of Unit Cell of the $La_{0.50}Li_{0.50}TiO_3$.

	Phase 1 $La_{0.5}Li_{0.5}TiO_3$)	Phase 2 ($LiTi_2O_4$)
a (Å)	3,8706 (4)	8,312 (5)
b (Å)	3,8706 (4)	8,312 (5)
c (Å)	7,785 (2)	8,312 (5)
Fraction (%)	94,67 (3)	5,3 (3)
V (Å^3)	116,64 (3)	574 (1)
Density (g/cm^3)	4,708	3,857

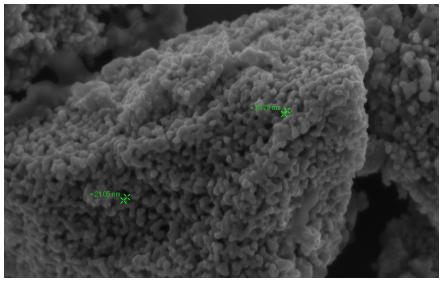

Figure 1: SEM-FEG micrographs of $La_{0,50}Li_{0,50}TiO_3$ powder obtained after heating at 700ºC/3h. Magnification: 177.390x.

References

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