Sub-micrometer $LiMn_2O_4$ Prepared by a Sol-gel, Spray-Drying Method

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Spinel $LiMn_2O_4$ has received great attention as the cathodic material for rechargeable lithium ion batteries because of its low cost and low toxicity. $LiMn_2O_4$ powder is usually prepared by solid-state reaction and sol-gel method.[1,2] In this paper a sol-gel, spray-drying method is introduced for the preparation of sub-micrometer $LiMn_2O_4$ powder.

A gel precursors was prepared by use of 1M LiOH and 1M $Mn(CH_3COO)_2$ solutions (with molecular ratio of Li : Mn = 1 : 2). The dry precursor powder was produced via a spray-dry process using the equipment as described in Fig. 1. Adjusting the rotating speed of the sprinkler could control the size of the precursor powder particles. After a heat treatment in air atmosphere the dry precursor could be transformed into $LiMn_2O_4$ powder. In the case of electrochemical measurements, the working electrode was composed of 80% $LiMn_2O_4$, 15% acetylene black, and 5% PTFE binder. The electrolyte was 1M $LiPF_6$ in EC:DMC = 1:1solution.

FTIR, XRD, DTA and TGA experiments provide the evidence that $LiMn_2O_4$ can be prepared at the temperature of 250ºC, 650ºC, 700ºC and 800ºC. The sample formed at 700ºC shows the best characteristics of spinel $LiMn_2O_4$ structure. Fig.2 shows the morphology of $LiMn_2O_4$ powder prepared by use of rotating speed 15000 rpm and heat treatment temperature 700ºC. This SEM image reflects the $LiMn_2O_4$ particles are in sub-micrometer size with the diameter of about 100nm-200nm.

For the samples synthesized at 250ºC, 650ºC, 700ºC and 800ºC, all of the voltammograms demonstrate the electrochemical characteristics of spinel $LiMn_2O_4$. Among them the sub-micrometer sized $LiMn_2O_4$ powder formed at 700ºC presents the best performance with well separate two pairs of peaks at 3.95V/4.08V and 4.05V/4.20V and highest peak current. In the heat treatment temperature rang of 250ºC - 550ºC the discharge capacity of samples increases with the increase of heating temperature. When the temperature rises further the capacity remains almost stable. However, the highest capacity of 128 $mAhg^{-1}$ occurs for the sample formed at 700ºC. The results of cycle life test for the samples synthesized at different temperatures are presented in Fig. 3. Compared with these curves, the sub-micrometer $LiMn_2O_4$ sample prepared using rotating speed 15000 rpm and heat temperature of 700ºC presents the best performance with highest discharge capacity and stability.

The results indicate that the sol-gel, spray-drying method may be used in industrial-scale to produce sub-micrometer sized $LiMn_2O_4$ for the use in lithium ion batteries.

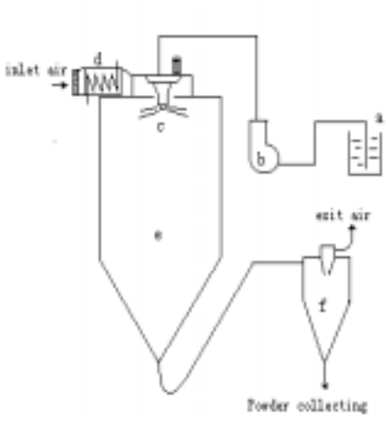

Fig.1 Schematic overview of spray dryer equipment: a- gel precursor container, b-pump, c- sprinkler, d- heater, e- spray-dry vessel, f- cyclone separator.

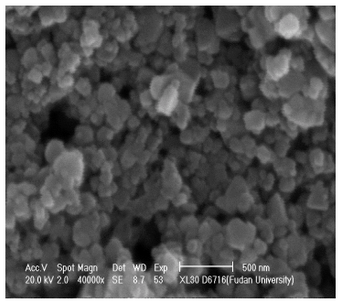

Fig.2 SEM morphology of $LiMn_2O_4$ powder synthesized at 700ºC

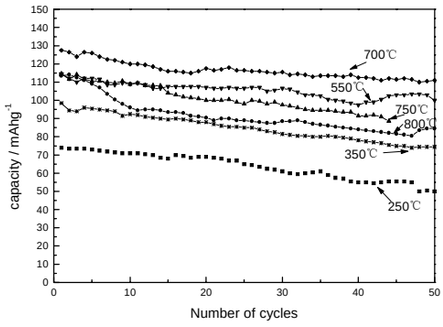

Fig.3 The variations of cycle discharge capacity of $LiMn_2O_4$ synthesized at different temperature, i = 0.2$mAcm^{-2}$.

References

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