

PREPARATION OF LiMn_2O_4 THIN FILMS FROM SUSPENSIONS BY ELECTROSTATIC SPRAY DEPOSITION (ESD) TECHNIQUE

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The fabrication of LiMn_2O_4 thin film electrodes has received considerable attention in recent years due to its application as cathode materials in a solid-state microbatteries. Until now, the techniques that are frequently used to synthesize thin film electrodes are chemical vapor deposition, sol-gel method, thermal vacuum evaporation, and pulse laser deposition techniques. Recently, Shoonman *et al.* proposed a novel technique that can prepared thin film electrodes, which is named electrostatic spray deposition (ESD) technique.¹ The ESD technique has advantages of simple and low cost set-up, high deposition efficiency, easy control of the composition and surface morphology compared to other techniques.

Up to now, the ESD method used a precursor solution to deposit thin films on a heated substrate, and the thin films are synthesized by chemical reaction of precursor solutions. However, the thin films synthesized by chemical reaction of precursor solutions and additives may leave some chemical residuals or impurities in the thin films, which may not be suitable for some cases where high-purity thin films are needed. Further, as-deposited films usually need to be heat-treated.

In the present study, we tried to apply ESD technique to fabricate electrodes for electrochemical quartz crystal microbalance (EQCM) technique. However, the heat treatment temperature after deposition by ESD technique, which is in the range of 600~800 °C, is beyond the temperature of a-b phase transition of quartz crystal used for EQCM techniques (573°C), and it restricted the use of the technique to EQCM measurement.

In searching for a new micro-fabrication technology for thin film cathodes at low temperature, we demonstrate the feasibility of using ESD method to produce thin films from a suspension containing LiMn_2O_4 fine particles. The advantage of using suspensions, instead of chemically prepared solutions is that the preparation does not introduce chemical reactions and any chemical by-products, and the as-deposited films usually do not need further heat treatment at high temperature. Therefore, the maximum temperature during fabrication can be limited as low as 400°C.

To fabricate LiMn_2O_4 thin film cathode from suspensions of fine particle LiMn_2O_4 , the key step is to generate fine suspension droplet with narrow size distribution. In order to effectively disperse LiMn_2O_4 fine particles and to produce a colloidally stable suspension, the choice of solvents is very important. Therefore, we considered various factors that can affect the stability and distribution of suspension when selecting a solvent-additive-dispersant system, which are particle charge, solubility of the additives, chemical compatibility of components, and viscosity and conductivity of the suspension. By considering above factors, we successfully prepared suspension of LiMn_2O_4 fine particle and fabricated LiMn_2O_4 thin film electrode from the suspension by ESD technique. Fig. 1 and 2 show XRD pattern and cyclic voltammogram of LiMn_2O_4 thin film prepared by ESD technique using the suspension. More details will be discussed at the meeting.

REFERENCE

1. C.H. Chen, A.A.J. Buysman, E.M. Kelder, J. Shoonman, *Solid State Ionics*, Vol. 80, pp 1, (1995)

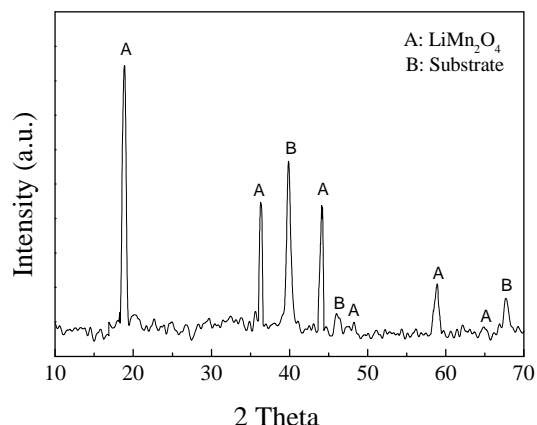


Fig. 1. XRD patterns of LiMn_2O_4 thin film deposited on Pt-coated Si wafer substrate by ESD technique using suspension of LiMn_2O_4 fine particles.

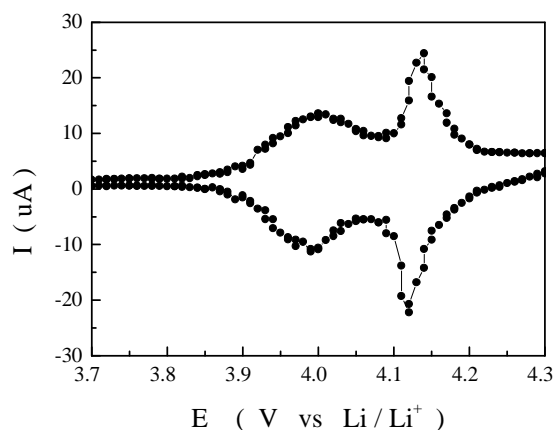


Fig. 2. The cyclic voltammogram of a LiMn_2O_4 thin film electrode prepared by ESD technique using suspension of LiMn_2O_4 fine particles. Scan rate; 0.2 mV/s, electrolyte; 1 M LiClO_4/PC .