

Synthesis and Characterization of Cathode Material for Li-ion Batteries

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Introduction

Lithium iron phosphate with the ordered olivine-type structure has attracted extensive attention due to low cost, safety and high compatibility with environment [1]. However its electronic conductivity ($\sim 10^{-9}$ S/cm) is very low, which leads to slow initial capacity loss and slow diffusion of Li⁺ ion across the LiFePO₄/FePO₄ boundary [2]. Therefore, many efforts have been devoted to increase the electrical conductivity which include (i) coating with a conductive materials [3]; (ii) ionic substitution [4], (iii) synthesis of particles with well-defined morphology and nano size particles [5] etc. Here we report the electrochemical behavior LiFePO₄-MWCNTs composite cathode material as reported at synthesized by tailoring the synthesis process.

Experimental Procedure and Synthesis

MWCNTs (SkyspringNanomaterials, USA) were soaked in concentrated nitric acid and ultrasonicated for 8 h. After this, the mixture was centrifuged, washed with distilled water and acetone until the PH was about 7. Finally the drying was done 80°C. The LiFePO₄ was prepared by reacting the stoichiometric amounts of Li₂CO₃, NH₄H₂PO₄ and FeC₂O₄.2H₂O. Before the reaction, the all the precursor powders were intimately mixed through a wet ball milling process for 12h in pure acetone using planetary type ball mill. After drying the solution, pellets were made which were then calcined at 350°C for 3 h in high vacuum. After calcination, the pellets were reground and functionalized MWCNTs were added from 5% (weight %). The pellets were again made and sintered at 600°C for 10 h in high vacuum to obtain LiFePO₄-MWCNTs composite. The pure was also prepared with the same synthesis route without the addition of MWCNTs.

Phase purity and crystal structure of LiFePO₄-0%MWCNTs and LiFePO₄-5%MWCNTs composite

were analyzed by powder X-ray diffraction (XRD) using Cu K α radiation in the 2 θ range of 10-70. The size and morphology of powder particles were determined with the help of field emission scanning electron microscope (FE-SEM, Philips, XL30 FEG, Eindhoven, Netherlands). Galvanostatic charge/discharge test was performed on WonA Tech (WBCS 3000) in the range of 2.0 to 4.9 V. For electrochemical tests, cathode were prepared by mixing the active material (LiFePO₄-MWCNTs), carbon black and Polyvinylidene fluoride (PVDF) in weight ratio of 75 : 15 : 10 respectively by adding suitable amount of N-methyl-2-pyrrolidone(NMP). After, mixing at 75°C, the slurry was cast on an aluminum foil using a doctor blade. The NMP was evaporated at 110°C for two hours followed by punching of electrodes of desired size. The coin type cells were assembled in an argon filled glove box using pure lithium foil as anode using celgard 2400 as a separator. One molar LiPF₆ in ethylene carbonate (EC) and diethyl carbonate (DEC) was used as electrolyte.

Results and Discussion

Fig.1 shows the XRD patterns for pure LiFePO₄ and LiFePO₄-5%MWCNTs composite.

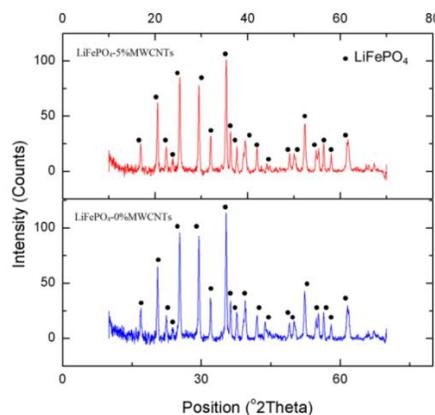


Fig.1. The XRD patterns for pure LiFePO₄ and LiFePO₄-5%MWCNTs composite samples.

The pattern for LiFePO_4 -5%MWCNTs can be indexed to single-phase material which is the same as the LiFePO_4 . The peaks for MWCNTs are not visible as due to overlapping with the main phase i.e LiFePO_4 . In addition, the amount of MWCNT is also low to be revealed easily.

Fig.2 shows the SEM image of LiFePO_4 and LiFePO_4 -5%MWCNTs composite. Flaky type particles with the average particle size of about 200 nm are observed for pure LiFePO_4 as shown in Fig.2a. In addition, the networks of MWCNTs in LiFePO_4 -5%MWCNTs composite can also be seen which can improve the electrical conductivity and thud the electrochemical performance of LiFePO_4 .

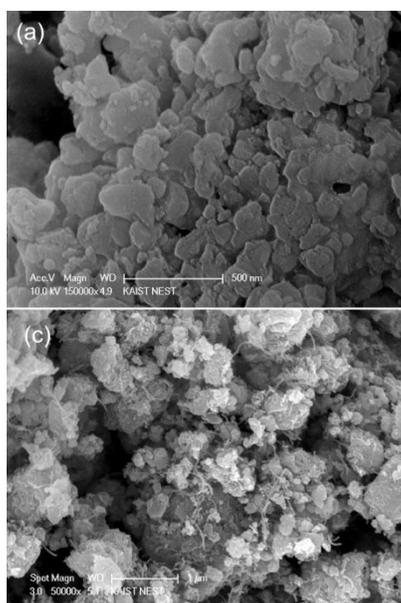


Fig.2. (a) SEM images showing morphology of pure LiFePO_4 , (b) LiFePO_4 -5%MWCNTs Composite

Fig.3 compares the initial galvanostatic charge/discharge profiles for LiFePO_4 -0%MWCNTs and LiFePO_4 -5%MWCNTs. It is clearly visible from the profiles that there is considerable improvement in the charge/discharge capacity of our synthesized for LiFePO_4 -5%MWCNTs composite. The discharge capacity has been increased from 122mAh g^{-1} to about 145mAh g^{-1} when 5%MWCNTs were added into LiFePO_4 . This increase in the discharge capacity can be attributed to wiring by MWCNTs which enables active material to transport lithium ions and

electron mass at fast rate. In addition, the wiring of MWCNTs may have also improved the contact area and thus superior electrochemical behavior. This is also consistent with the previous results [6].

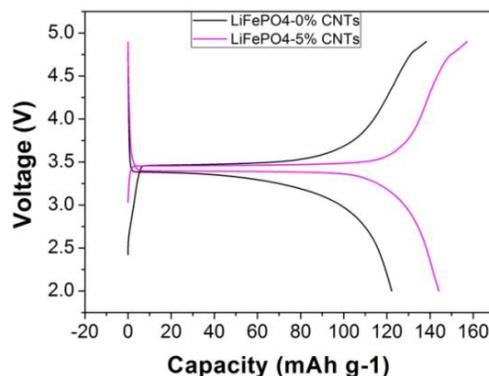


Fig. 3. Shows the initial galvanostatic charge/discharge profile for LiFePO_4 -0%MWCNTs and LiFePO_4 -5%MWCNTs at C/10 rate.

Conclusion

Pure LiFePO_4 and LiFePO_4 -MWCNTs composite were successfully synthesized as phase pure and well crystalline. Addition of MWCNTs didn't result in any impurity phase formation and didn't change the phase structure of LiFePO_4 . The LiFePO_4 -5%MWCNTs composite exhibits superior electrochemical behavior when compared with the pristine LiFePO_4 . This improvement in the electrochemical behavior can be attributed to the network wiring of MWCNTs which may have resulted in the increase in the electrical contact area and thus high electronic conductivity. LiFePO_4 -5%MWCNTs composite can be used as a cathode material for Li-ion batteries.

References:

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