

The effect of heat preservation time on the electrochemical properties of LiFePO₄

Rui He ^{1, 2, a}, Lihui Zhang ^{1, 2, b, *}, Xue Bai ^{1, 2, c}, Zhenfa Liu ^{1, 3, d}

¹Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang, Hebei Province

²Hebei Engineer Research Center for Water Saving in Industry, Shijiazhuang, Hebei Province

³School of Chemical Engineering, Hebei University of Technology, Tianjin, P.R. China

^akxyherui@163.com, ^bzlhkxy@sohu.com,

^cbxuesmile0910@126.com, ^dlzf63@sohu.com

*Corresponding author email : kxyherui@163.com

Abstract. LiFePO₄ was prepared via high temperature solid-state method at different heat preservation time. XRD and SEM was used to test the structure and morphology of LiFePO₄. Land 2001 was used to test the electrochemical performance of LiFePO₄. The results illustrated that well-crystallized LiFePO₄ composite with homogeneous small particles was obtained by XRD and SEM. And the optimum heat preservation time was 4 hour. From charge/discharge test, it can be seen that at 0.2C, LiFePO₄ has initial discharge capacities of 159.1mAh/g at the heat preservation time 4 hour. From the rate capacity, it can be seen that the discharge capacity was of optimum sample remains above 99% after 200 cycles.

Keywords. Lithium iron phosphate, heat preservation time, electrochemical properties.

1. Introduction

In recent years, the research and development of the lithium ion battery anode material have attracted much attention in industrial countries because of its high energy density, long lasting and many more [1-3]. LiFePO₄ is one of the most commonly used cathode material. It is low cost and very environmental protection. And it also has high security, excellent cycle life and high thermal stability [4-5]. In 1997, Goode-nough et al. reported for the first time that Lithium iron phosphate was reversible in the intercalation and DE intercalation of lithium ions. It is considered to be one of the most promising cathode materials of the dynamic lithium ion batteries [6]. Thackeray [7] considered that the discovery of LiFePO₄ making the arrival of a new era of lithium ion batteries.

In this paper, the materials of LiFePO₄ were prepared via high temperature solid-state method. The effects of different heat preservation time on structure and performance of LiFePO₄ were studied. Through this method, the materials of LiFePO₄ with good electrochemical performance and cycle performance were prepared.



2. Experiment

LiFePO₄ compounds were synthesized via high-temperature solid-state method. The stoichiometric amounts of LiH₂PO₄ (99.5%), LiAc (99.5%), FeC₂O₄·2H₂O (99.5%) and C₆H₆O₃·2H₂O were mixed in ethanol and ball milled at the speed of 300r/min for 4h. Then the mixture was dried at 80°C in the oven for 5h. After drying, the mixture was claimed at 700°C for 4h, 6h, 8h and 10h, respectively under N₂ atmosphere. The heating rate was 3°C/min. The vacuum tube furnace (HEFEI KEJING materials technology co. LTD) was used for the preparation of composite electrode materials.

The purity and structure of the material were detected with X-ray diffraction (XRD) (Ultima IV, Rigaku) using Cu K α radiation. Scanning electron microscopy (SEM) (Inspect S50 FEI) was used to observe the particle morphology. Laser particle size analyzer (Rastarizer 3000, Malvern Instruments Ltd.) Electrochemical measurements were carried out using Land CT2001A (Land, Wuhan blue electric technology co., LTD China).

3. Results and discussion

Fig. 1 shows the XRD pattern of LiFePO₄ prepared at different heat preservation time. The XRD patterns of at different heat preservation time samples are similar to the standard diffraction peaks of LiFePO₄ (PDF 40-1499). The XRD pattern reveals a pure LiFePO₄ phase without any other impurities. The XRD pattern demonstrates that the pure phase material was prepared by high temperature solid-state method.

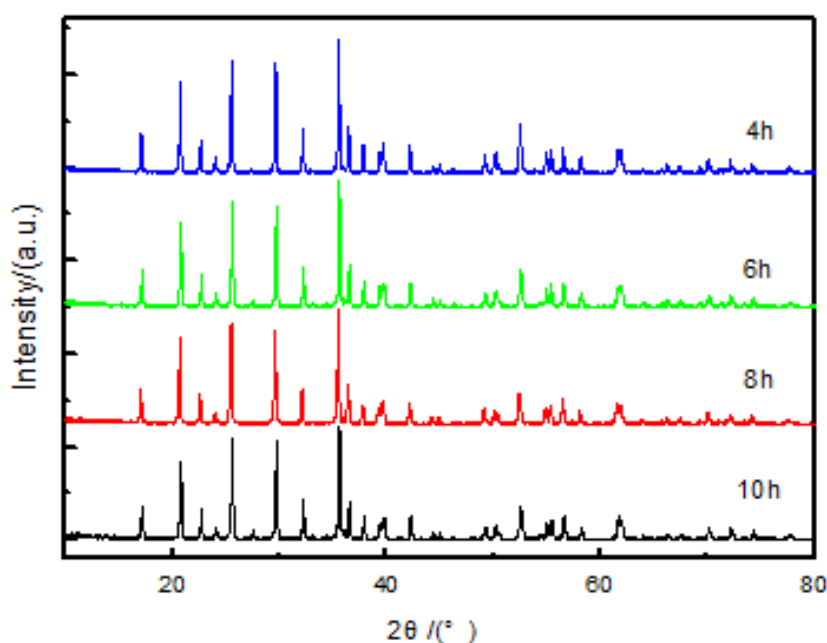


Fig. 1 XRD patterns of LiFePO₄ prepared at different heat preservation time

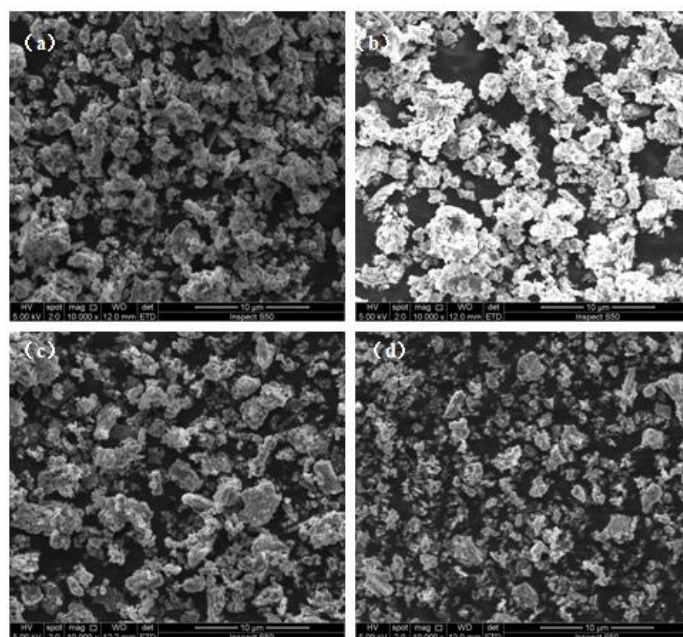


Fig. 2 SEM patterns of at different heat preservation time

Fig. 2 show the SEM images of materials synthesized at different heat preservation time. Fig. 2(a) is the holding time of 4h. Fig. 2(b) is the heat preservation time of 6h. Fig. 2(c) is the heat preservation time of 8h. Fig. 2(d) is the heat preservation of 10h. The particle size basically no change. And there is no clearly boundary between particles. From Fig. 2, it can be seen that the particle distribution is relatively more uniform at Fig. 2(a) than others. And the dispersion of the particle is better than Fig. 2(b), (c) and (d).

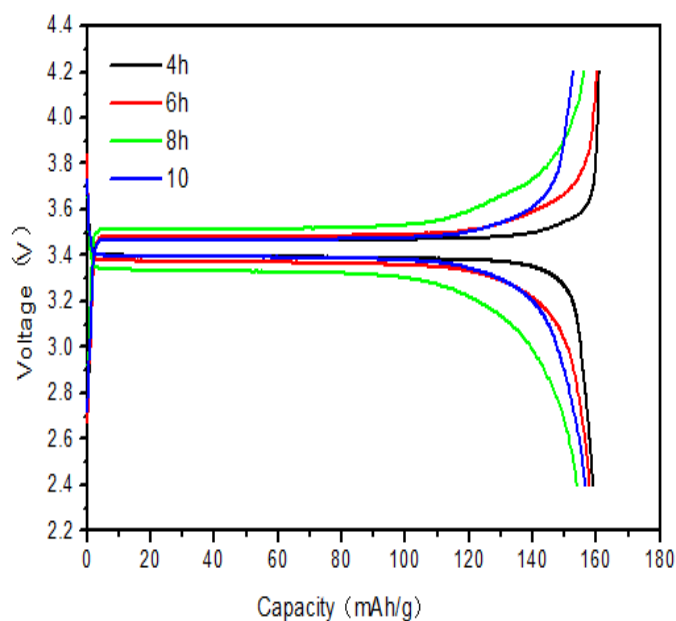


Fig.3 charge/discharge of at different heat preservation time

Fig. 3 shows the initial charge and discharge curves of LiFePO_4 in the range of 2.4V to 4.2V at 0.2C. And there is a clear platform at 3.4V in Fig. 3. The initial

specific discharge capacity at 0.2C is 159.1, 158, 156 and 154mAh/g when the heat preservation time is 4h, 6h, 8h and 10h respectively. The optimum heat preservation time of the material is 4h.

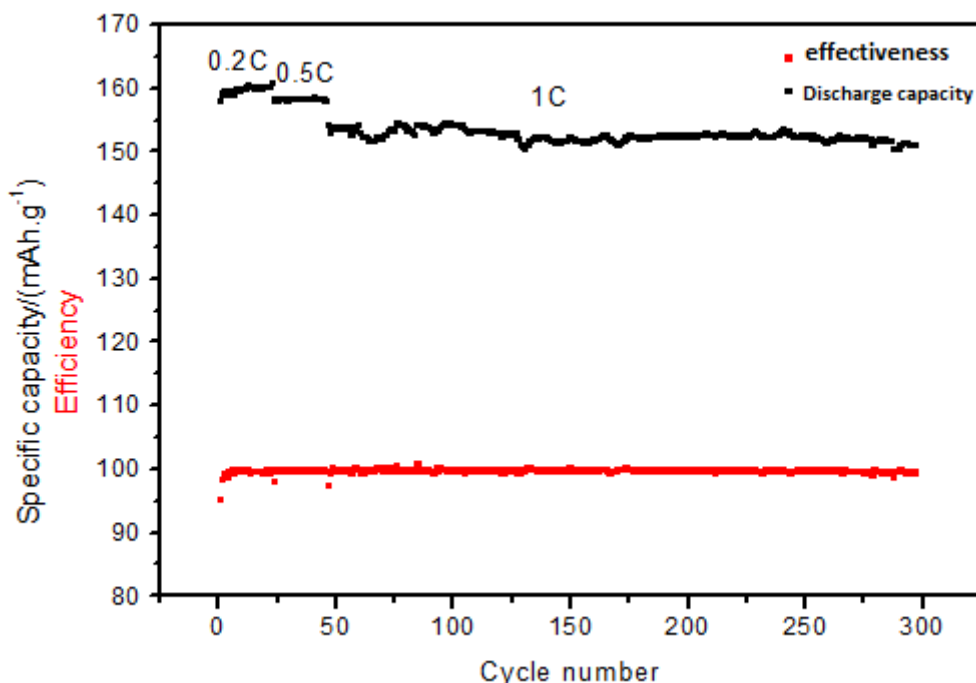


Fig. 4 cycle performance of the optimum sample

Fig. 4 shows the cycle performance of the heat preservation time at 4h. From the image, it can be seen that the first discharge capacity is 157.8mAh/g, 158mAh/g and 154mAh/g at 0.2C, 0.5C and 1C, respectively. The discharge capacity of the sample is above 150mAh/g. The discharge capacity remain kept 151 mAh/g after 200 cycles. The capacity retention rate is 98%.

4. Conclusion

LiFePO₄ cathode material was successfully synthesized by High temperature solid phase method. The impact of different heat preservation time on the microstructure and electrochemical performance of the material were studied. Particle distribution is becoming more and more uniform when the heat preservation is 4h. And the heat preservation of 4h is the best holding time. The discharge capacity remain kept 151 mAh/g after 200 cycles. The capacity retention rate is 98%.

Acknowledgements

This work was financially supported by the Hebei Science and Technology Support Project (16294403D).

References

- [1] Padhi A K, Nanjundaswamy K S, Goodenough J B. Phospho-olivines as positive-electrode materials for rechargeable lithium batteries. *Journal of the electrochemical Society*, 1997, 144(4): 1188-1194.
- [2] Zhang Ddongyun, Zhang Peixin, Lin Muchong, Property and Structure of Carbon-coated LiFePO₄. *Journal of Inorganic Materials*, 2011, 26(3); 265-270.
- [3] Chen G Y, Song X Y, Richardon T J. Electron microscopy study of the LiFePO₄ to FePO₄ phase Transition. *Electrochemical and Solid-State Letters*, 2006,9(6):A295-A298.

- [4] A.S. Anderesson. B. Kalska. L. Häggström, J.O. Thomas., J. Solid State Ionics 130(1-2)(2000)41.
- [5] J. Wang and X. Sun: Understanding and recent development of carbon coating on LiFePO_4 cathode materials for lithium-ion batteries. J. Energy Environ. Sci. 5, 5163(2012).
- [6] Tarascon J M, Armand M. [J]. Nature, 2001,414:359-367.
- [7] Thackeray M. [J]. Nature Mater, 2002, 1 :81-82.