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Li₃VO₄: an insertion anode material for magnesium ion batteries with high specific capacity



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ABSTRACT

Li₃VO₄ (LVO) is a promising insertion-type anode material for lithium ion batteries (LIBs) while its electrochemical performance in magnesium ion batteries (MIBs) is rarely reported. Here, mesoporous LVO/Carbon (LVO/C) hollow spheres are synthesized by a facile spray-drying method and their electrochemical performance as Mg^{2+} insertion-host material is investigated for the first time. Galvanostatic charge-discharge results of LVO/C show no obvious platform in the potential range of $0.5\sim2.5 V$ vs. Mg^{2+}/Mg . The LVO/C delivers a high discharge capacity of 318 mAh g⁻¹ at first cycle and exhibits good cycle performance. The electrochemical intercalation process is proved by element mapping, *ex*-situ X-ray Powder diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) analysis. Our results show that LVO is a promising anode material for MIBs.

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1. INTRODUCTION

LIBs are facing severe challenges in energy density, safety and price, which greatly limit their applications, especially in electric vehicles and grid energy storage [1]. Many efforts have been devoted to develop alternative energy storage devices with high volumetric energy density, low price and better safety, such as sodium-ion batteries [2]. Among them, rechargeable magnesium (Mg) batteries have attracted increased attention as one of postlithium batteries [3]. Compared to Li, Mg is much more abundant and evenly spread on the earth's crust, which are beneficial for reducing the production cost of rechargeable Mg batteries [4]. The divalent nature provides Mg a higher volumetric energy density (Mg 3832 mAh cm^{-3} vs. Li 2046 mAh cm^{-3}). Additionally, Mg is dendrite free and less sensitive in atmosphere, making it a safe anode in rechargeable Mg batteries [5]. However, the incompatibility of Mg anode with conventional electrolyte (such as Mg (ClO₄)₂ and Mg(TFSI)₂ in nonaqueous polar organic solvents) hinders its development [6]. Although Mg can be efficiently dissolution/deposition in various organohaloaluminates [(MgR₂)_x- $(AlCl_{3n}R_n)_v]$, recent studies showed that these electrolyte are air sensitive, highly volatile and provide a limited electrochemical

http://dx.doi.org/10.1016/j.electacta.2017.06.143 0013-4686/© 2017 Elsevier Ltd. All rights reserved. window, which are unfavourable to develop high-voltage Mg batteries [7]. An alternative choice to overcome this obstacle is to develop MIBs by replacing Mg anode with intercalation anode material that are compatible with conventional electrolyte [8,9]. Until now, only several types of anode materials have been reported. Sn [10,11], Pb [12], and Bi [13] attract much attention due to their low working voltage and high theoretical capacity. For example, Pb formed Mg₂Pb when used as anode material, corresponding to a theoretical capacity of 2300 Ah/L, and delivered a low voltage ($\sim 125 \text{ mV}$ vs. Mg) [12]. Guo group reported that spinel Li₄Ti₅O₁₂ nanoparticles showed an initial discharge capacity of 70 mAh g^{-1} in pure Mg electrolyte [14,15]. Recently, Chen group developed layered Na2Ti3O7 nanoribbons for reversible electrochemical Mg storage with a theoretical capacity of 88 mAh g^{-1} [8]. Although many efforts have been devoted to promote the development of anode material for MIBs, the cycle ability or incompatibility of these materials with the conventional electrolyte still need to be improved. Therefore, exploring new types of anode material is highly needed.

 Li_3VO_4 is a promising insertion anode material for LIBs with excellent rate performance due to its high ionic conductivity [16]. The corner-shared VO₄ and LiO₄ tetrahedrons form hollow lanternlike three dimensional structure (Fig. 1a), providing empty sites and intercalation channels for Li⁺ [17]. However, the large band gap (3.9 eV) gives poor electronic conductivity to this material [18]. In order to obtain better electrochemical performance, carbon was

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Fig. 1. (a) Crystal structure of orthorhombic Li₃VO₄; (b) XRD pattern of the prepared LVO/C.

usually used as an effective additive to improve electrical conductivity [19,20]. On the other hand, decreasing the particle size of active materials or constructing hollow-structured materials to shorten diffusion lengths of Li⁺ are also effective ways [21,22]. Spray drying method is an economical and rapid process to obtain spherical particles from nano to micron sizes that are agglomeration-free [23]. It is also a convenient way to incorporate carbon into the composite. Recently, our group has successfully synthesized

mesoporous LVO/C hollow spheres by spray drying method with superior rate capability and cyclic stability for LIBs [24]. Since Mg²⁺ (72 pm) and Li⁺ (76 pm) have similar ionic radius, the channels suitable for Li⁺ transport in LVO may also be fit for Mg²⁺. Therefore, it is of great interest to examine the electrochemical performance of LVO in MIBs.

In this work, we investigated electrochemical performance of mesoporous LVO/C hollow spheres prepared by spray-drying



Fig. 2. SEM image (a), TEM image (b) and HRTEM image (c) of the prepared LVO/C; (d) N₂ adsorption-desorption isotherms and pore size distribution (inset) of the prepared LVO/C.

method as an insertion-anode material for MIBs. As we expected, the LVO shows good electrochemical performance in MIBs. A high discharge capacity of 318 mAh g^{-1} can be obtained at first cycle. The insertion process can be confirmed by element mapping, *exsitu* XRD and XPS results.

2. EXPERIMENTAL SECTION

2.1. Material synthesis

The LVO/C was prepared as we previously reported [24]. In detail, 2 mmol V_2O_5 and 6 mmol Li_2CO_3 were completely dissolved in 70 mL deionized water under continuous stirring. Then 0.9 g glucose was added into the solution and the solution was stirred for half an hour. The resulting clear solution was sprayed using the Buchi mini spray drier B-290 in N₂ atmosphere. The inlet and outlet temperature were 200 °C and 113 °C, respectively. The precursor was collected and calcined in Ar atmosphere at 550 °C for 5 h at the heating rate of 5 °C min⁻¹.

2.2. Characterizations

The morphology of LVO/C were examined with the field emission scanning electron microcopy (SEM, HITACHI S-4800). TEM images were investigated by high-resolution transmission electron microscopy (HRTEM, FEI TECNAI G2 F30). XRD was performed on the Rigaku MiniFlex600 X'pert diffractometer (D8 advance, Japan) using Cu K α radiation (λ = 1.5405 Å). XPS was conducted on PHI Quantum 2000 Scanning ESCA Microprobe. The nitrogen adsorption-desorption isotherms were attained on an ASAP 2020 Micropore Analyzer. The carbon content of the LVO/C was analyzed by elemental analyzer (Vario-EL-III).



Fig. 3. (a) Typical charge/discharge curves at the 1st, 5th and 10th cycle of LVO/C at 20 mA g^{-1} ; (c) The dQ/dV curves of first cycle of LVO/C. All specific capacity is based on the mass of LVO/C.

2.3. Electrochemical measurements

A slurry composed of 70 wt% of prepared LVO/C, 20 wt% of acetylene black and 10 wt% of polyvinylidene fluoride (PVDF) was spread on titanium foil and dried in vacuum oven at 80 °C over night. Electrochemical performance was evaluated with CR2032 coin cell, a 2-electrode configuration, using freshly polished Mg foil as counter electrode. Celgard 2400 were used as separator and $0.5 \text{ M Mg}(ClO_4)_2$ /acetonitrile (AN) was used as electrolyte. Anhydrous acetonitrile (CH₃CN, Alfa Aesar, 99.8+%) was dried with 3 Å molecular sieves for 48 h before used. The water content was < 10 ppm measured by Karl Fischer titration. Anhydrous magnesium perchlorate (Mg(ClO₄)₂, Aladdin) was used as received. However, the water content could not be measured accurately by Karl Fischer titration due to the interference from the strong oxidant $Mg(ClO_4)_2$. Galvanostatic charge-discharge test was conducted in the potential range of $0.5 \sim 2.5 \text{ V}$ vs. Mg²⁺/Mg at 20 mAg^{-1} (0.034C, 1C = 592 mAg⁻¹) at 25 °C. For *ex*-situ XRD test, the cells were discharged to 0.7 V, 0.5 V and charged to 1.5 V, 2.0 V and 2.5 V, respectively. For XPS test, the cells were discharged to 0.5 V and charged to 2.5 V. All cells were disassembled in glovebox and the electrodes were washed with AN several times then dried at room temperature. For ex-situ XRD test, electrodes were sealed with protective film in glove box before taken to XRD test. For XPS test, the electrodes were sealed with laminated aluminum film (used for LIBs, Fig. S1) before taken out of the glove box. The electrodes were well sealed in Ar atmosphere before taken out of bag and sent to XPS specimen chamber.

3. RESULTS AND DISCUSSION

Considering the low electronic conductivity of pristine LVO, the mesoporous LVO/C hollow spheres with glucose as carbon source were chosen as the studying object. Fig. 1b is the XRD pattern of the prepared LVO/C. All the diffraction peaks are in accordance with an orthorhombic Li₃VO₄ phase (JCPDS No. 038-1247). The morphology and microstructure of the LVO/C were studied by SEM and TEM. SEM image (Fig. 2a) and TEM observation (Fig. 2b) indicate that LVO/C are hollow spheres with diameter of about $0.5 \sim 5$ micrometres. HRTEM (Fig. 2c) result reveals that LVO particles are coated with a thin carbon layer. The interplanar distance of LVO is about 0.41 nm, which is agree with the d-spacing of (110) planes of orthorhombic LVO. The carbon content measured by elemental analysis is 26%. N₂ adsorption-desorption isotherms of the prepared LVO/C present a type-IV curve. The pore size distribution curve calculated by Barrett-Joyner-Halenda (BJH) method indi-



Fig. 4. Cycle performance of the LVO/C at 20 mAg^{-1} .



Fig. 5. (a) TEM image of LVO/C after discharge to 0.5 V; (b) HAADF image of a single LVO/C sphere after discharge to 0.5 V; (c - f) Element mapping of C, V, O and Mg elements in a single LVO/C sphere, respectively.

cates that the LVO/C has mesoporous structure with a narrow pore diameter distribution at about 3.9 nm. The specific surface area of the LVO/C as measured by the N₂ absorption Brunauer–Emmett–Teller (BET) method is 126.5 m² g⁻¹. The large specific surface area is beneficial for electrolyte accessibility and shorten the diffusion path of Mg²⁺ in solid material, thus improve Mg²⁺ insertion kinetics [24,25].

The LVO/C exhibits good electrochemical performance. The galvanostatic charge-discharge test was executed at $0.5 \sim 2.5$ V vs. Mg²⁺/Mg and typical charge-discharge curves are shown in Fig. 3a. Both the discharge and charge curves show no obvious platform. The LVO/C delivers the discharge capacity of 318 mAh g⁻¹ and the charge capacity of 242 mAh g⁻¹ at the first cycle. The low initial coulombic efficiency (76%) may be attributed to the phenomenon that some of the inserted Mg²⁺ were trapped in the host material [26]. Compared to its theoretical capacity capacity (592 mAh g⁻¹), only 53.7% of the theoretical capacity can be achieved in the initial

discharge process. The low utilization of LVO may be caused by the strong coulomb interaction between Mg²⁺ and LVO host [1,3,25]. It is worth noted that the discharge capacity of the LVO/C is much higher than $Li_4Ti_5O_{12}$ (70 mAh g⁻¹) and $Na_2Ti_3O_7$ (88 mAh g⁻¹). The discharge and charge curves of the 5th and the 10th cycle are similar to the first cycle, indicating the electrochemical behaviors of Mg²⁺ inserting into and extracting from the LVO/C during cycling are similar. The differential capacity (dQ/dV) curves computed from the galvanostatic discharge-charge profiles give us a better understanding of the insertion process. The dQ/dV curve of the first discharge process (Fig. 3b) indicates that intercalation of Mg²⁺ most occurs between $0.5 \sim 1.2 \, \text{V},$ while the result of Mg^{2+} extraction during charge process shows a peak at 1.25 V and a broad peak between 1.5~1.9 V. Apart from the high capacity, the LVO/C also exhibits good cycle performance (Fig. 4). At the 10th and the 15th cycle, the LVO/C can still deliver 195 mAh g^{-1} and



Fig. 6. Ex-situ XRD of the LVO/C electrode at the first discharge-charge process; (b) Enlargement of ex-situ XRD.

171 mAh $\rm g^{-1},$ corresponding to the capacity retention of 61% and 54%, respectively.

The TEM (Fig. 5a) and High-angle annular dark field (HAADF) (Fig. 5b) images of the LVO/C after first discharge to 0.5 V reveal that the LVO/C sphere is maintained well after discharge process. Element mapping images of the latter LVO/C sphere are also shown in Fig. 5(c~f). The homogenous distribution of C, V, O and Mg, indicates the existence of Mg^{2+} in active materials after discharge process.

To study the structural reversibility of the LVO/C during insertion/extraction process of Mg²⁺, ex-situ XRD was conducted on the LVO/C electrodes at first cycle (Fig. 6). The electrodes were sealed in protective film to prevent the impact of O_2 in air (Fig. S2). For each case, no extra peaks appear under the test condition, indicating an intercalation mechanism of the LVO for Mg²⁺, which is similar to that in Li batteries [27]. At open circuit voltage (OCV), diffraction peaks of LVO are clearly observed. When discharged to 0.7 V, peaks are weaker and almost all peaks disappear at the end of discharge process. During the charge process, diffraction peaks gradually enhanced but become weaker and broader compared to those at OCV, which may be caused by the decrease of LVO particle size and structure failure of LVO during Mg²⁺ ions intercalation process [27,28]. It is worth noted that peak of LVO/C at 21.50° (110 plane) slightly shift to left after discharge process and do not shift back after charge process (Fig. 6b), indicating the increase of (110) plane d-spacing of original structure due to the Mg²⁺ ions insertion [17]. The decrease of peak intensities and increase of d-spacing of (110) plane provide us a proof for Mg²⁺ ions insertion process. Exsitu XRD results reveal the insertion of Mg²⁺ into LVO is not completely reversible, which is agree with the discharge-charge results. One possible reason for this is that the extraction of Mg²⁺ from LVO is difficult due to its divalent nature and Li⁺ in Li₃VO₄ may extracted from structure during charge process [8,14]. Further

study to find out the reason and improve its structure reversibility is in progress.

XPS was utilized to further prove the insertion of Mg^{2+} into LVO. Similar to Li⁺, the intercalation of Mg^{2+} into LVO is accompanied with the reduction of V⁵⁺ to V⁴⁺, V³⁺ and even lower valence. Fig. 7 shows the high resolution photoelectron spectra of V2p_{3/2} of three different state electrodes, that is fresh, fully discharged to 0.5 V and charged to 2.5 V, respectively. After discharge process, peak become broader at lower binding energy, indicating the reduction of V⁵⁺ to lower valences. After charge to 2.5 V, peak gets recovery, suggesting the oxidation of low states of V during the extraction process of Mg²⁺. The fitted curves of V2p_{3/2} give the clarification of valence of V. For the fresh electrode

(Fig. 7a), peak at 517.1 eV corresponds to V⁵⁺ [29]. A small peak at 515.8 eV can be ascribed to V^{3+} , which may come from the reduction of V⁵⁺ by glucose during the heat treatment [29]. After discharge process (Fig. 7b), three peaks at 517.6 eV, 516.7 eV and 515.2 eV can be fitted, corresponding to V^{5+} , V^{4+} and V^{3+} , respectively. Upon charge (Fig. 7c), two peaks at 517.3 eV and 516.0 eV correspond to V^{5+} and V^{3+} , respectively. Peaks for fresh and charge electrode are little different, indicating little structure difference of the two states due to the insertion/extraction of Mg²⁺, which is also agreed with the *ex*-situ XRD results. Table S1 displays the assignments and atomic% of different valence of V for fresh, discharged and charged electrode. The calculated capacity based on XPS results is 179 mAh g⁻¹, much lower than the discharge capacity (318 mAh g^{-1}) . The main reason for this is that the discharged electrode, especially the surface of the electrode, may has been reoxidized by O_2 because of its high reactivity [30,31]. Since XPS is a surface analysis technique (< 10 nm), the information we got just from the surface of the electrode, not the body part. In this case, the calculated specific capacity based on XPS results will be lower than the discharge capacity.



Fig. 7. XPS spectra of V2p_{3/2} of LVO/C at (a) OCV, (b) discharge to 0.5 V and (c) charge to 2.5 V, respectively.

4. CONCLUSIONS

In conclusion, the LVO/C was firstly studied as Mg^{2+} insertion anode material. The insertion process can be proved by the element mapping, *ex*-situ XRD and XPS results. The LVO/C delivers a high specific capacity of 318 mAh g⁻¹ at first cycle and maintains 57% capacity at the 15th cycle. The unique structure of mesoporous LVO/C hollow spheres with high specific surface area shorten the diffusion path of Mg^{2+} in solid material, thus improve Mg^{2+} insertion kinetics effectively. In addition, the preparation of LVO/C by spray-drying method is simple and producible. Our encouraging results show that LVO is a potential anode material for MIBs.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j. electacta.2017.06.143.

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