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## Nanoplates-stacked baguette-like LiVO<sub>3</sub> as high performance cathode materials for lithium-ion batteries

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LiVO<sub>3</sub> has been proposed as an appealing cathode material to substitute traditional capacitylimited cathodes for high energy lithium-ion batteries. However, just like the other Li-V-O compounds, the application of LiVO<sub>3</sub> cathode is restricted by its poor cycling stability and rate capability. In this paper, we report, for the first time, nanoplates-stacked baguette-like LiVO<sub>3</sub> by an in situ template method. In this unique structure, the micro-sized baguette shape can help to inhibit the dissolution of vanadium in comparison with nanosized LiVO<sub>3</sub>, and the stacked nanoplates can provide a short diffusion pathway for lithium ions. Benefiting from these advantages, this material can deliver a very high capacity of 275.6 mAh g<sup>-1</sup> with excellent cycling stability, Moreover, a capacity of 156.1 mAh g<sup>-1</sup> can be obtained up to 4 C (1200 mA g<sup>-1</sup>). The exceptional electrochemical performance suggests the use of nanoplates-stacked baguette-like LiVO<sub>3</sub> as alternative cathode materials for high-energy lithium-ion batteries. Particularly, the synthesis strategy demonstrated herein may be extended to fabricate other types of multifunctional vanadium-based compounds for energy storage.

#### Introduction

In recent years, the demand of lithium ion batteries to power the next generations of communication devices (5G), electric vehicles (EVs) and hybrid electric vehicles grows rapidly. However, the practical lithium storage capacities for the current commercialized cathodes, including LiCoO<sub>2</sub>, LiMn<sub>2</sub>O<sub>4</sub> and LiFePO<sub>4</sub>, are limited to 150 mAh g<sup>-1</sup> due to their intrinsic structural characteristics.<sup>1-3</sup> Therefore, in order to further increase the energy density of LIBs, many efforts have been made to develop alternative cathode materials, such as Li-rich layered composites,<sup>4-7</sup> metal fluorides,<sup>8-10</sup> polyanionic compounds,<sup>11, 12</sup> et. al. Among the numerous candidates, Li-V-O compounds seem to be attractive cathode materials because of their high theoretical capacities, abundant resources and good safety features.

In the Li-V-O compounds family, LiV<sub>2</sub>O<sub>5</sub> and LiV<sub>3</sub>O<sub>8</sub> were found electro-active for lithium storage very early and received considerable attentions. Orthorhombic LiV<sub>2</sub>O<sub>5</sub> was reported to be able to deliver a high discharge capacity of > 300 mAh g<sup>-1</sup>, but the lithium intercalation process involves two successive redox reactions,  $V^{5+}/V^{4+}$  and  $V^{4+}/V^{3+}$ , which may lead to structural transition and capacity fade.<sup>13, 14</sup> In contrast, monoclinic LiV<sub>3</sub>O<sub>8</sub> undergoes only one redox reaction ( $V^{5+}/V^{4+}$ ), and thus exhibits good structural reversibility during cycling. However, LiV<sub>3</sub>O<sub>8</sub> cathode is still far from large-scale application which is probably because the electrochemical performances of the cathode are strongly related to the morphology and synthesis method.<sup>15-18</sup> In this context, researchers turned to explore other available Li-V-O compounds. Recently, Pralong firstly demonstrate that LiVO<sub>3</sub> can serve as a high-capacity cathode for lithium-ion battery, the uni-dimensional character of its structure enable it totally reversible during lithium intercalation and de-intercalation process.<sup>19</sup> Later, Tu and his co-workers successively prepared LiVO<sub>3</sub> compounds by two different methods, and further investigated their structures and electrochemical properties.<sup>20, 21</sup> As we can see, the LiVO<sub>3</sub> cathodes reported in their works can exhibit a high discharge capacity of > 250 mAh g<sup>-1</sup>, whereas the cycling performance is still unsatisfactory. Apparently, the dissolution of vanadium and irreversible phase transformation during cycling should take the main responsibility for the capacity decay.

On the basis of the above consideration, we have developed an in situ template method to prepare nanoplatesstacked baguette-like LiVO<sub>3</sub>. In this novel structure, the microsized baguette shape can help to inhibit the dissolution of vanadium and ensure an excellent cycling stability, the nanoplates can provide a short diffusion pathway and facilitate fast lithium ion transportation. As a result, the nanoplatesstacked baguette-like LiVO<sub>3</sub> delivers a high discharge capacity of 275.6 mAh g<sup>-1</sup> with capacity retention of 86.8% after 100 cycles. Moreover, a capacity of 156.1 mAh g<sup>-1</sup> can be obtained even at a rate of 4 C (1200 mA g<sup>-1</sup>), such superior

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electrochemical performance enables it to be a feasible cathode material for high performance lithium-ion batteries.

#### **Experimental Section**

#### **Sample Preparation**

Baguette-like LiVO3 was prepared with an in situ template method using triblock copolymer F127 and phenolic resin as the framework. The resol precursor (Mw <500) was prepared according to the literature method and was dissolved in ethanol to form a 20 wt % ethanolic solution.<sup>22</sup> In a typical preparation, 1.65 gram of triblock copolymer F127 ( $M_w$ =12600, PEO106PPO70PEO106, Sigma-Aldrich) was first dissolved in ethanol, then 0.02 mol CH<sub>3</sub>COOLi (99%) was added in and dissolved. At the same time, 0.02 mol NH<sub>4</sub>VO<sub>3</sub> and oxalic acid were dissolved in ethanol to afford a clear solution, and then the clear solution was added into the former one and stirred for 1h at 40 °C. Later, resols' ethanolic solution was added subsequently. After being stirred for 2 h, the mixture was transferred into large petri dishes. The mixture was left overnight to evaporate ethanol and then heated for 24 h at 100 °C in an oven for thermal polymerization. The precursor was grounded into fine powders, pressed into pellets. Calcination was carried out sequentially, first at 400 °C for 4 h and next at 500 °C for 6 h in air to get baguette-like LiVO<sub>3</sub>.

The control sample (nano-sized LiVO<sub>3</sub>) was prepared by a polymer-pyrolysis method according to previous literature. The polymeric precursor was synthesized by dissolving stoichiometric CH<sub>3</sub>COOLi, NH<sub>4</sub>VO<sub>3</sub> and oxalic acid in acrylic acid and copolymerizing the acrylates by adding a small amount of initiator in the solution at continuous stirring at 80 °C. The as formed polymeric precursor was firstly dried at 80 °C, and then calcined under the same condition as the baguette-like LiVO<sub>3</sub>.

#### Characterizations

The crystalline structure of the as-prepared baguette-like LiVO<sub>3</sub> was characterized by X-ray diffraction (XRD, Bruker AXS, D8 Advance). The morphologies of the LiVO<sub>3</sub> were characterized by scanning electron microscopy (SEM, Histachi S-4800) and transmission electron microscopy (TEM, JEM-2100F).

#### **Electrochemical measurements**

Electrochemical testing of the baguette-like LiVO<sub>3</sub> material was performed using coin cells with lithium metal as a counter electrode. The electrolyte was 1 M LiPF<sub>6</sub> dissolved in a mixture of ethylene carbonate (EC), dimethyl carbonate (DMC) and ethylene methyl carbonate (EMC) (1:1:1 by wt), and the separator was a microporous membrane (Celgard 2400). The cathode was prepared by mixing 80 wt % LiVO3, 10 wt % Super P and 10 wt % PVDF together and dissolving the electrode mixture into N-methyl-2-pyrrolidone (NMP) to form homogeneous slurry, then coating the electrode slurry on aluminum foil. 2016 type coin cells were assembled in an

Journal of Materials Chemistry A Accepted Manuscri spectroscopy (EIS) was recorded using the impedance measuring unit (IM 6e, Zahner) with oscillation amplitude of 10

argon-filled glove box and galvanostatically charged and discharged within the voltage range of 1.0 V to 3.5 V at room temperature. To measure the quantity of vanadium dissolution in the electrolyte, both test cells (baguette-like LiVO3 and control sample) after 100 cycles were disassembled in an argon-filled glove box, and then the two electrodes were taken out and immersed into EC. After stirring for homogenization, the quantity of vanadium in the liquid phase was measured. Vanadium content was determined with a Varian (liberty II axial view) inductively coupled plasma-atomic emission spectrometer (ICP-AES), the wavelength and detection limit for vanadium were 292.402 nm and 5 µg L<sup>-1</sup>, respectively. The rate capability was assessed at room temperature with constant charge rate of 0.1 C and various discharge rates from 0.1 C to 4 C, where 1 C corresponds to 300 mA g<sup>-1</sup>. Cyclic voltammetric measurement was also carried out with the three-electrode cell

at the scan rate of 0.1 mV s<sup>-1</sup>. Electrochemical impedance

mV in the frequency range of 100 kHz to 0.01 Hz.

#### **Results and discussion**

The nanoplates-stacked baguette-like LiVO<sub>3</sub> was prepared by constituent coassembly of triblock copolymer F127, resols and salts precursors, the synthesis process can be schematically illustrated in Fig. 1. Firstly, the phenolic resols, lithium and vanadium salts in the solution would attach to the hydrophilic blocks of the triblock copolymer and form cross-linked framework. Driven by the minimization of the total energy of the system and the interaction force between the hydrophobic PPO blocks, the small primary units begin to bind together. As a result, the hydrophilic groups, including EO chains, resols and salts, would be self-assembled side by side in an ordered fashion to form an alternating layered structure. After the removal of surfactant at elevated temperature, the decomposed



Fig. 1 Schematic illustration of the formation process of nanoplates-stacked baguette-like LiVO<sub>3</sub>.

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Fig. 2 Rietveld refined XRD pattern (blue) of nanoplates-stacked baguettelike  $LiVO_3$  with experimental data (red) and difference curve (olive). The standard XRD pattern for  $LiVO_3$  is placed at the bottom for comparison.

intermediates will remain uniformly distributed in the hydrophilic layer, and thus grow into nanoplates. Upon further calcination at much higher temperature, the nanoplates tend to stack together and form baguette-like LiVO<sub>3</sub>.

Fig. 2 shows the X-ray diffraction (XRD) pattern and its Rietveld-refined pattern for the as obtained baguette-like LiVO<sub>3</sub>. As can be seen, the XRD pattern can be assigned to wellcrystallized monoclinic LiVO<sub>3</sub> (JCPDS Card No.: 33-0835, space group: C2/*c*). The lattice parameters calculated from XRD using Rietveld refinement are a = 10.156 Å, b = 8.429 Å, c = 5.883 Å and = 110.47° respectively, which are quite consistent with previous reports.<sup>19</sup> For comparison, the structure of the control sample (nano-sized LiVO<sub>3</sub>) was also tested. As can be seen in Fig. S1, the XRD pattern of the control sample is almost the same as the baguette-like LiVO<sub>3</sub>, but the diffraction peaks appear much broader, implying a decrease in its crystalline size.

The morphological features of the nanoplates-stacked baguette-like LiVO3 were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Fig. 3a shows a SEM image of the as-prepared LiVO<sub>3</sub> material, baguette-like particles with a length of 10 µm, a width of 2-6 µm and a thickness of ~4 µm are observed. In the highmagnification SEM image as shown in Fig. 3b, it is interesting to observe that the baguette-like LiVO3 is composed of numerous stacked plate units from the end of the particles. This unique structure is further confirmed by Fig. 3c, which shows the image of the baguette-like LiVO<sub>3</sub> from side view. We can observe distinctly many plates with a few tens of nanometers thickness for each individual one and these nanoplates stack on top of each other to form baguette-like particles. Locally magnified HRTEM image of baguette-like LiVO<sub>3</sub> (Fig. 3f) is shown to provide further insights into the crystalline structure of the as-prepared samples. Two legible lattice fringes with basal distances of 2.16 Å and 2.06 Å can be observed in Fig. 3f, which are consistent with the (-421) and (420) lattice spacing of



Fig. 3 SEM images (a-c), TEM images (d, f) and HRTEM image (e) of nanoplates-stacked baguette-like LiVO<sub>3</sub> (inset of SAED).

monoclinic LiVO<sub>3</sub>, respectively. A selected-area electron diffraction (SAED) pattern (inset of Fig. 3f) revealed that the nanoplate grows with selective orientation along the [100] direction, this microscopic image can in fact account for the significant increase of the (200) peak shown in Fig. 2. The morphology of the control sample is also characterized in Fig. S2, as can been, the LiVO<sub>3</sub> powders are composed of small particles with an average size of ~100 nm. It has been demonstrated that the dissolution of vanadium in electrolyte during cycling depends on the morphology of the Li-V-O compounds. Apparently, the surface activity of microsized baguette-like LiVO<sub>3</sub> is much lower than nanosized LiVO<sub>3</sub>, so the unique structure in this work should help to inhibit the dissolution of vanadium from the lattice, and thus enhance the cycling performance of LiVO<sub>3</sub> cathode. Moreover, the dynamic and thermodynamic characteristics of vanadium dissolution from  $LiVO_3$  are still unclear, the issue that whether the [100] growth orientation of the nanoplates is beneficial to the alleviation of vanadium dissolution needs further research.

Lithium insertion/extraction properties of the nanoplatesstacked baguette-like LiVO<sub>3</sub> were investigated by cyclic voltammetry (CV) and galvanostatic charge/discharge cycling. Fig. 4a shows typical CV curves of a LiVO<sub>3</sub> electrode at a scan rate of 0.1 mV s<sup>-1</sup>. The reduction process during the first cycle is different from the ones observed in subsequent cycles, suggesting a possible structure transition from the initial LiVO<sub>3</sub> structure to a more stable structure. During the positive scan, a main anodic peak appears at ~2.7 V, corresponding to the oxidation reaction of the V<sup>4+</sup>/V<sup>5+</sup> redox couple. After the first scan, the position of the main redox peaks shifts to higher potential, and no noticeable change of current or potential was observed for the couple peaks in the following cycles,

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**Fig. 4** Electrochemical characterization of the baguette-like LiVO<sub>3</sub> electrode: (a) cyclic voltammograms at a scan speed of 0.1 mV s<sup>-1</sup>, (b) the charge-discharge curves at a current density of 0.1 C (1 C = 300 mA g<sup>-1</sup>).

indicating that the lithium insertion/extraction process is highly reversible and the crystalline structure of the cathode keeps stable after the structural transition in the initial discharge process. Furthermore, the CV curves for all the cycles exhibit at least three pairs of quite symmetrical small redox peaks, implying a complex multiphase transition mechanism during Li-ion insertion and extraction processes. The structural transition in the first cycle can also be observed through the charge/discharge curves in Fig. 4b. When tested at a constant current of 0.1 C (1 C = 300 mA  $g^{-1}$ ) in the potential range of 1.0 to 3.5 V (vs. Li/Li<sup>+</sup>), one plateau, located around 1.7 V, was distinctly observed for the baguette-like LiVO<sub>3</sub> electrode in the initial discharge, suggesting the lithium intercalation process involves a biphasic reaction. After the first discharge, the potential curves for both charge and discharge process become sloped, indicative of a monophasic mechanism. The chargedischarge profiles are in good agreement with the CV results in Fig. 4a. Interestingly, both the average charge and discharge potentials increased gradually from the 1<sup>st</sup> cycle to the 50<sup>th</sup> cycle, which might be associated with the electrochemically active process of LiVO<sub>3</sub>. However, the gap between the average

charge and discharge potentials for the  $100^{\text{th}}$  cycle grew wider compared to that for the  $50^{\text{th}}$  cycle, meaning that the resistance increased after 100 cycles, which might be attributed to the dissolution of vanadium during cycling.

Cycling performances and the corresponding coulombic efficiencies of the baguette-like LiVO<sub>3</sub> electrode are plotted in Fig. 5a. As can be seen, the cathode can deliver an initial discharge capacity of 275.6 mAh g<sup>-1</sup> with capacity retention of 86.8% after 100 cycles. ICP measurements revealed that the vanadium dissolution ratio of the electrode is only 2.2% after 100 cycles. In comparison, this value increased to 9.7% for the control sample (nanosized LiVO<sub>3</sub>). As a result, although nanosized LiVO<sub>3</sub> can also exhibit a high discharge capacity of 277 mAh g<sup>-1</sup>, only 55.4% of its initial capacity can be retained after 100 cycles (Fig. S3). Obviously, this excellent cycling stability of baguette-like LiVO<sub>3</sub> electrode originated from its smaller surface area for decreasing the irreversible surface chemical reactions. From the typical charge-discharge curves in Fig. 4b, we can find that the average working voltage is around 2.5 V, thereby a high energy density of 692.5 Wh kg<sup>-1</sup> (apart from the weight of electrolyte, current collector, package, etc.) is obtained, which far exceeds current commercialized



**Fig. 5** (a) Cycling performance and the corresponding coulombic efficiencies (charge/discharge) of the baguette-like LiVO<sub>3</sub> electrode at a current density of 0.1 C, (b) discharge capacity of baguette-like LiVO<sub>3</sub> as a function of charge/discharge cycles at different discharge current densities of 0.1 C, 0.2 C, 1/3 C, 0.5 C, 1 C, 2 C and 3 C (charge at 0.1 C).

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LiFePO<sub>4</sub>/C (462 Wh kg<sup>-1</sup>) and LiMn<sub>2</sub>O<sub>4</sub>/C (440 Wh kg<sup>-1</sup>) batteries. The coulombic efficiency of the electrode reached 91% during the first cycle and further increased to about 100% subsequently. To the best of our knowledge, this material exhibits the best performance in terms of high capacity and excellent cycling stability for Li-ion insertion compared to the LiVO<sub>3</sub> cathodes reported so far.<sup>19-21</sup> The rate capability of baguette-like LiVO<sub>3</sub> electrode was also evaluated in Fig. 5b. The electrode delivers reversible capacities of 259 mAh g<sup>-1</sup> at 0.5 C, 232 mAh  $g^{-1}$  at 1 C, and 156 mAh  $g^{-1}$  at 4 C. It can be seen that although the particle size of baguette-like LiVO<sub>3</sub> reaches microscale level, the rate capability is still superior to that of nanosized  $\text{LiVO}_3$  reported in previous work.  $^{20,\ 21}$  The superior rate capability of our cathode should be mostly attributed to the nanoplates-stacked structure, in which the thin nanoplate unit and large interspaces between the nanoplates are favorable for Li ions to pass through, and thus leading to a fast kinetics of the nanoplates-stacked LiVO<sub>3</sub>.

To gain further insight into the electrochemical mechanisms operating for the baguette-like LiVO<sub>3</sub> electrode, EIS measurements of the cathodes were conducted at the end of the 1<sup>st</sup>, 10<sup>th</sup>, 50<sup>th</sup> and 100<sup>th</sup> cycle, respectively. As shown in Fig. 6, all four Nyquist plots display a semicircle at high frequency and a sloped line at low frequency, corresponding to the charge transfer resistance at electrolyte-electrode interfacial (R<sub>ct</sub>) and the lithium diffusion Warburg impedance within the electrode, respectively. By comparing the diameters of the arcs, we found that the charge-transfer resistance of baguette-like LiVO<sub>3</sub> gradually decreased from the 1<sup>st</sup> cycle to the 50<sup>th</sup> cycle, implying an activation process and enhanced reversibility of the electrode. Subsequently, the diameter of the semicircle increases slightly when going from the 50<sup>th</sup> electrode to that subjected to 100<sup>th</sup> cycle, showing a minor increase in R<sub>ct</sub> as the electrode goes through long cycles, possibly due to the dissolution of vanadium. These observations may account for



Fig. 6 Electrochemical impedance spectra (EIS) of the baguette-like  $LiVO_3$  electrode at different cycle.



Fig. 7 Ex-situ XRD patterns of the baguette-like LiVO<sub>3</sub> electrode at various depths of charge-discharge.

the average charge/discharge potential variations upon cycling in Fig. 4b.

To further convince the cycling stability of the electrode, we explored XRD analysis to characterize the structural changes of the electrode. Fig. 7 shows the ex-situ XRD patterns of the baguette-like LiVO<sub>3</sub> electrode at different depths of charge and discharge. As is shown, after first discharge at 1.5 V, the diffraction peaks of the product are completely different from that of the pristine phase. One XRD peak emerged at 15.1°, probably characterizing the formation of Li<sub>2</sub>VO<sub>3</sub> phase.<sup>19</sup> Furthermore, three small peaks located at 41.7°, 43.1° and 44.1° can also be observed in the pattern, and these three peaks always existed during cycling, meaning that some unknown Li-V-O species without lithium-storage activity were generated due to the structure transition after initial discharge. These phenomena may account for the initial capacity loss observed from the baguette-like LiVO<sub>3</sub> electrode in Fig. 4b. After first charge at 4.0 V, the XRD signals of Li<sub>2</sub>VO<sub>3</sub> phase disappeared. Instead, one XRD peak emerged at 14.3°, right at the same position as the (110) peak for the pristine phase LiVO<sub>3</sub>, but much weaker and broader, indicating that monoclinic LiVO<sub>3</sub> transformed to a more stable amorphous LiVO<sub>3</sub> phase after initial cycle. Interestingly, the XRD patterns at the 2<sup>nd</sup> cycle, 10<sup>th</sup> cycle and 50<sup>th</sup> cycle are almost the same as the pattern after 1<sup>st</sup> cycle (charge to 4.0 V), implying a structural stability of the electrode during cycling. These results do demonstrate that the nanoplates-stacked baguette-like LiVO<sub>3</sub> can keep stable structure during repeated charge-discharge process, and thus promoting long-term stable cycling.

#### Conclusions

In summary, nanoplates-stacked baguette-like LiVO<sub>3</sub> have been synthesized by an in situ template method. In this unique structure, the micro-sized baguette shape can help to inhibit the dissolution of vanadium compared to nanosized LiVO<sub>3</sub>, and the stacked nanoplates can provide a short diffusion pathway for lithium ions. Benefiting from these advantages, this material can deliver a very high capacity of 275.6 mAh g<sup>-1</sup> with excellent cycling stability, a capacity of 156.1 mAh g<sup>-1</sup> can be obtained up to 4 C. The exceptional electrochemical performance suggests the use of nanoplates-stacked baguette-like LiVO<sub>3</sub> as alternative cathode materials for high-energy lithium-ion batteries. In addition, the synthesis strategy demonstrated herein may be extended to fabricate other types of multifunctional vanadium-based compounds for energy storage.

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#### Notes and references

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 $\dagger$  Electronic Supplementary Information (ESI) available: the XRD pattern, SEM image and electrochemical peformance of the control sample (nanosized LiVO<sub>3</sub>) are included. See DOI: 10.1039/c000000x/

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#### TOC



Nanoplates-stacked baguette-like LiVO<sub>3</sub> demonstrates a high capacity of 275.6 mAh g<sup>-1</sup> with stable cycling and superior rate capability