

High-volumetric-capacity WSe₂ Anode for Potassium-ion Batteries^①

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ABSTRACT Exploring high-capacity electrode materials is critical for the development of K-ion batteries. In this work, we report a layered-structured tungsten selenide (WSe₂) anode, which not only delivers an ultrahigh volumetric capacity of 1772.8 Ah/L (or 188.4 mAh/g) at a current density of 5 mA/g but also exhibits good rate capability (72 mAh/g at 200 mA/g) and cycling stability (83.14% capacity retention over 100 cycles at 100 mA/g). We have also revealed the underlying reaction mechanism through ex situ X-ray powder diffraction. Furthermore, proof-of-concept full-cell batteries comprising of WSe₂ anodes and Prussian Blue cathodes are capable of delivering an energy density of 135.2 Wh/kg_{cathode+anode}. This work highlights the potential of WSe₂ as a promising high-volumetric-capacity anode material for rechargeable potassium-ion batteries.

Keywords: tungsten selenide, potassium-ion batteries, high volumetric capacity, conversion reaction;

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1 INTRODUCTION

Lithium-ion batteries (LIBs) have been widely used in portable electronics and electric vehicles because of their high energy and power density^[1, 2]. However, large-scale applications of LIBs for energy storage are seriously limited by the scarce reserves and uneven distribution of lithium resources^[3]. Given the earth-abundant potassium resources and a similarly low redox potential of K/K⁺ as Li/Li⁺ (−2.93 V and −3.04 V, vs standard hydrogen electrode), potassium-ion batteries (KIBs)^[4, 5] have been recognized as promising alternatives or supplements to LIBs for large-scale energy storage system^[6, 7]. Despite tremendous potential, the development of KIBs has been largely hindered by the lack of suitable high-capacity electrode host materials. The (de)intercalation of the large-size K⁺ ions often results in obvious structural degradation and therefore short cycle life to the electrode materials. On the other hand, the large ionic

size typically leads to sluggish K⁺-diffusion kinetics within the electrode materials and undesired fast-charging capability. Therefore, it is critical to search for new host materials that can not only store a large amount of K⁺ ions reversibly but also exhibit fast reaction kinetics.

Layered-structured transition metal dichalcogenides (TMDs), such as MoS₂^[8] and WS₂^[9], have received considerable attention as promising KIBs anode materials as they operate based on conversion-type reactions and are capable of delivering high theoretical capacities^[10, 11]. Very recently, Jiao *et al.* reported the anode behavior of a WSe₂/N-doped porous carbon composite (WSe₂/N-PC)^[12]. Differing from the MoS₂ and WS₂ anodes that exhibit flat voltage plateaus and relatively long cycle life, the WSe₂ nanocomposite exhibits a high capacitive-dominated K⁺-ion storage capacity without obvious voltage plateaus and limited cycle life. It is reasonably speculated the nanostructuring of WSe₂ is responsible for these discrepancies. Considering this,

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it is necessary to investigate the intrinsic K^+ -ion storage behavior of WSe_2 anode material and further improve its cycling stability.

Here we report a systematic investigation on the K^+ -ion storage behavior of the pure WSe_2 . We reveal that WSe_2 , serving as an anode material for KIB, operates at an average potential of 0.85 V (versus K/K^+) accompanied by a flat voltage plateau and delivers an ultrahigh volumetric capacity of 1772.8 Ah/L at a current density of 5 mA/g. Moreover, the WSe_2 anode exhibits good rate capability (72 mAh/g at 200 mA/g) and long-term cycling stability (112 mAh/g remained over 100 cycles at 100 mA/g). Ex-situ X-ray powder diffraction analysis reveals that WSe_2 anode first undergoes intercalation reactions and then follows reversible conversion reactions. We have further fabricated full-cell KIBs utilizing a WSe_2 anode and a Prussian Blue cathode. The proof-of-concept batteries deliver a high energy density of 135.2 Wh/kg_{cathode+anode}. WSe_2 may open new opportunities for the development of high-capacity electrode materials adopted for high-energy-density rechargeable potassium-ion batteries.

2 EXPERIMENTAL

2.1 Materials

WSe_2 powders (99.8% metals basis, CAS registry number: 12067-46-8) were purchased from Shanghai Macklin Biochemical Co., Ltd. Sodium carboxymethylcellulose (CMC) and Super-P carbon black were purchased from Hefei Kejing Materials Technology Co., Ltd. Battery grade potassium bis(trifluoromethylsulfonyl) imide (KTFSI) and diethylene glycol dimethyl ether (DEGDME, water content < 1 ppm) were purchased from DoDoChem. KTFSI was vacuum dried at 100 °C for 48 h before dissolving in DEGDME solvent.

2.2 Materials characterization

X-ray powder diffraction (XRD) patterns were collected on a Rigaku Ultima IV X-ray diffractometer with $CuK\alpha$ radiation source ($\lambda = 1.54184 \text{ \AA}$) at a 2θ range of $5\sim 80^\circ$ and a scanning rate of $1^\circ/\text{min}$. The operating voltage and current are 40 kV and 40 mA, respectively. The microscopic size and morphology of WSe_2 were characterized by a field-emission scanning electron microscope (FESEM, HITACHI SU-8010).

2.3 Electrochemical measurements

The WSe_2 electrode was prepared through a slurry blade-coating method. Specifically, the WSe_2 powders, carboxymethylcellulose (CMC) binder, and Super-P carbon black were

mixed homogeneously with a mass ratio of 8:1:1 in deionized water. The slurry was coated on an Al foil and dried under vacuum at 80 °C for 12 h. The mass loading of WSe_2 is $\sim 2.5 \text{ mg/cm}^2$. To investigate the electrochemical performance of WSe_2 electrode, the standard CR2032-type coin half cells were assembled with potassium metal foils as counter electrodes in an argon-filled glove-box. 5 M potassium bis(trifluoromethylsulfonyl)imide (KTFSI) in diethylene glycol dimethyl ether (DEGDME) was adopted as electrolytes^[13] and glass fibers (Grade GF/F, Whatman) were applied as separators. Galvanostatic charge/discharge cycling tests at different current densities were carried out on LANHE CT-2001A in the voltage range of 0.1 to 2 V (vs. K/K^+). Cyclic voltammetry (CV) was performed on an electrochemical workstation (Bio-Logic SP-300) at a scanning rate of 0.1 mV/s in the voltage range of 0.1 to 3 V (vs. K/K^+). All electrochemical tests were conducted at a constant temperature of 28 °C.

3 RESULTS AND DISCUSSION

As illustrated in Fig. 1a, WSe_2 exhibits a distinct layered structure with neighboring layers interconnected *via* Van der Waals force. The interlayer spacing of 6.49 Å is larger than those of ReS_2 ^[14], MoS_2 ^[8], WS_2 ^[9], $NbSe_2$ ^[15] and $ReSe_2$ ^[16] (Fig. 1b), and favors the intercalation of large-ionic-size K^+ ions^[17]. The crystal structure and phase purity are confirmed by X-ray powder diffraction (XRD). As shown in Fig. 1c, all the diffraction peaks could be accurately indexed to the standard WSe_2 structure (ICSD-40752) with space group $P63/mmc$ (No. 194). The distinct Bragg peaks at 13.62° , 31.40° , 32.17° , 37.80° , and 41.70° correspond to (002), (100), (101), (103), and (006) lattice planes, respectively, with a highly preferred orientation in the (002) lattice plane. Scanning electron microscopy (SEM) imaging reveals that WSe_2 exhibits hexagon plate-like morphology with a lateral size of about $\sim 100\sim 200 \text{ nm}$ and a thickness of $\sim 30 \text{ nm}$ (Fig. 1d).

The electrochemical behaviors of WSe_2 were explored by cyclic voltammetry (CV) and galvanostatic charge-discharge cycling tests. Fig. 2a shows the three-cycle CV curves collected at a scan rate of 0.1 mV/s within 0.01~3 V (vs. K/K^+). The irreversible reduction peak at $\sim 0.45 \text{ V}$ in the first cycle can be attributed to the formation of a solid electrolyte interphase (SEI)^[18, 19]. In the following cycles, three pairs of reduction/oxidation peaks appearing at

1.16/1.18, 1.6/2.0 and 1.91/2.68 V indicate reversible K⁺-ion insertion and extraction within WSe₂. Fig. 2b depicts the galvanostatic voltage profiles of WSe₂ at a current of 5 mA/g. WSe₂ delivers an initial discharging capacity of 297 mAh/g and a reversible charging capacity of 188.4 mAh/g as well as an initial Coulombic Efficiency (CE) of 61.9%. The irreversible capacity loss during the first cycle coincides with those observed in CV analysis and could be attributed to the formation of SEI, which is commonly observed in KIB anodes^[20-23]. The volumetric capacity is estimated to be as high as 1772.8 Ah/L according to a density of 9.39 g/cm³ for WSe₂, exceeding previously reported anode materials including graphitic carbon, organics, Ti-based compounds, metal sulfides as well as alloy-type P and Sn^[8, 9, 13, 15, 24-34] (Fig. 2c). Figs. 2b and 2d depict the long-term cycling performance of the WSe₂ anode at 5 mA/g. WSe₂ still delivers a specific capacity of 144.4 mAh/g after 50 cycles, corresponding to a capacity retention of 76.4%. Equally importantly, the flat voltage plateau remains during 50 cycles (Fig. 2d), demonstrating the high reversibility of this new

K⁺-WSe₂ electrochemistry. At a current density of 20 mA/g, the WSe₂ anode delivers a reversible capacity of 117.4 mAh/g after 100 cycles and an average CE of 98.68% (Fig. 2e). At even higher current densities of 50 and 100 mA/g, capacities of 101.7 and 112 mAh/g are achieved after 100 cycles (Fig. 2f), respectively, with capacity retention (70.6% and 83.14%) exceeding that for previously reported WSe₂/N-PC (64% over 100 cycles at 100 mA/g)^[12]. Moreover, the WSe₂ anode delivers a higher initial CE of 61.9% (versus 50.06% for WSe₂/N-PC).

Apart from good long-term cyclability, the WSe₂ anode also exhibits good rate capability. As shown in Figs. 2g and 2h, the WSe₂ anode delivers capacities of 183, 170, 151, 138, 107, 82, and 72 mAh/g at current densities of 5, 10, 20, 30, 50, 100, and 200 mA/g, respectively, and the voltage plateaus remain during rate-capability cycling. Equally importantly, the capacity recovers to 166 mAh/g when switching the current density back to 5 mA/g, manifesting the high reversibility of the WSe₂ anode against both deep and fast charging.

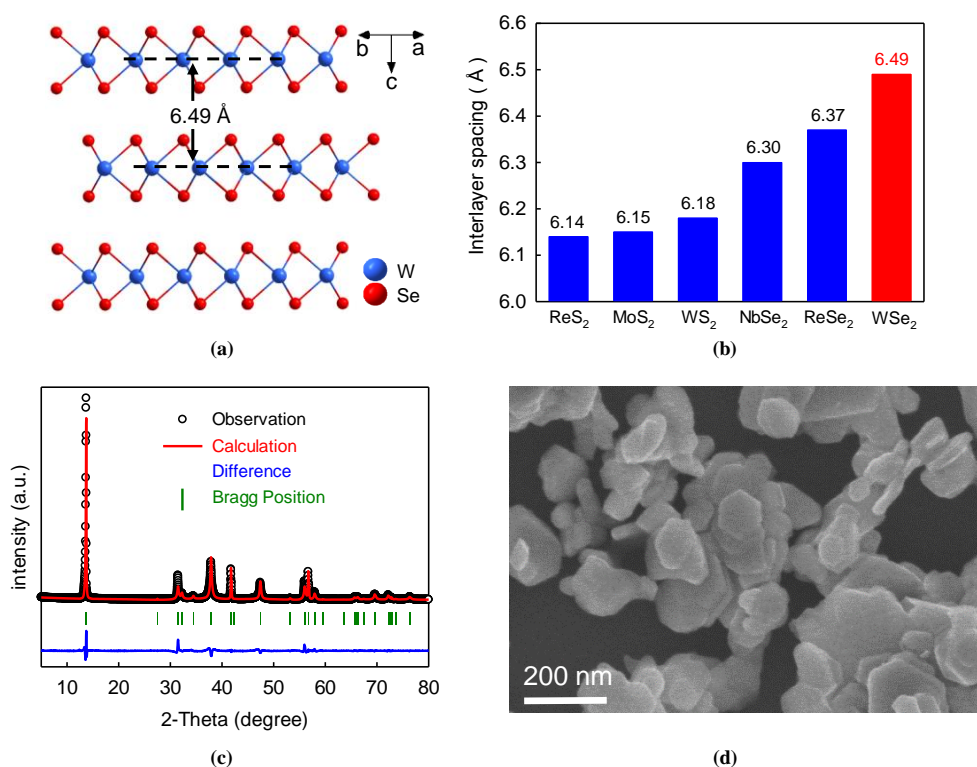


Fig. 1. (a) Crystal structure of WSe₂; (b) Comparison of interlayer spacing among TMDs; (c) Rietveld refinement of the XRD pattern of WSe₂ (R_{wp} = 9.69%, and R_p = 7.26%); (d) SEM image of WSe₂

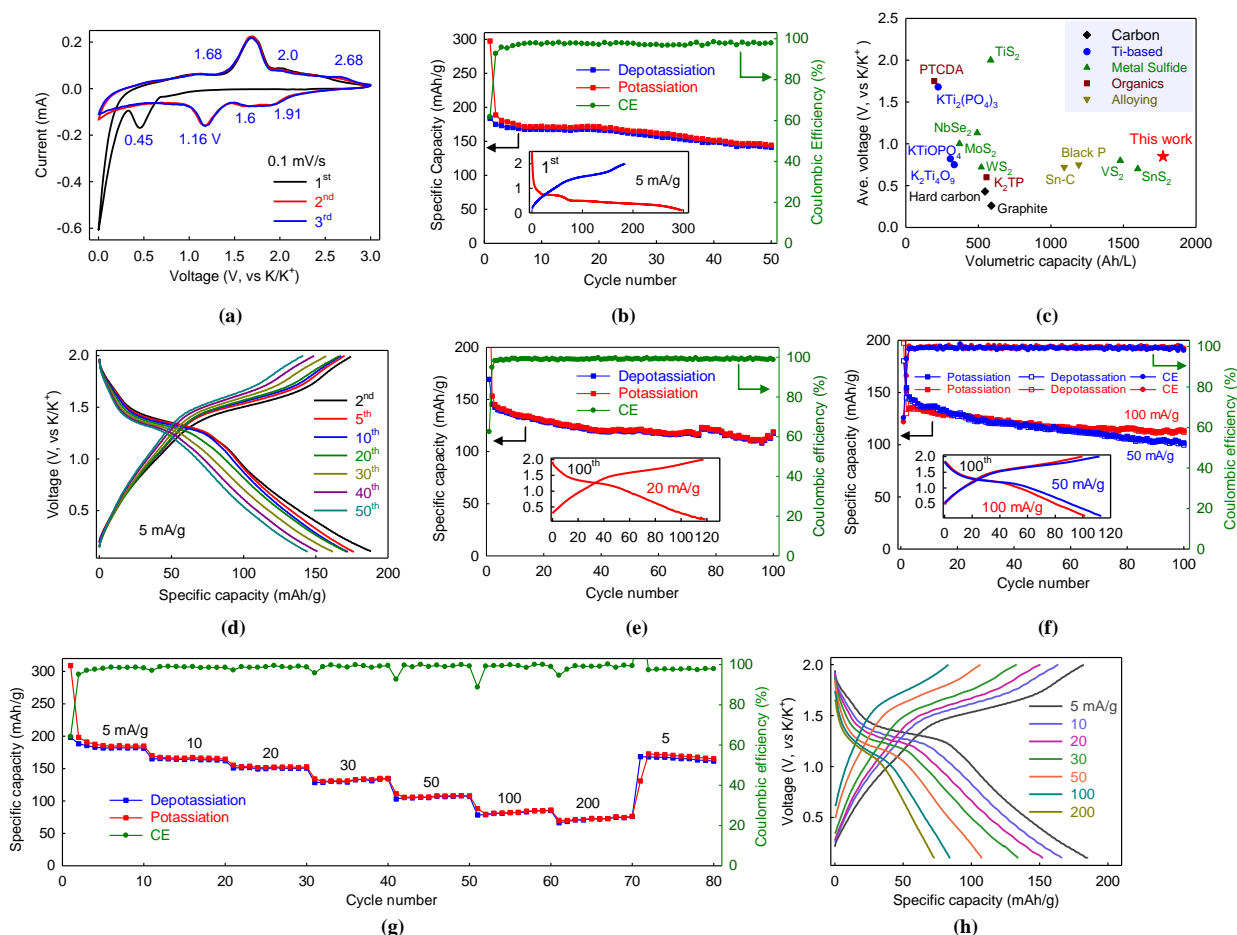
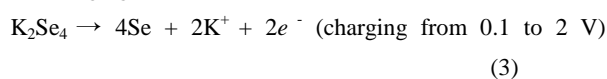
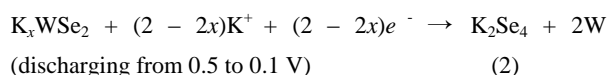
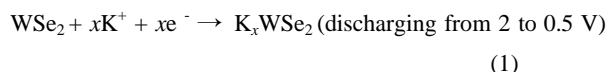


Fig. 2. (a) CV curves of the WSe₂ at 0.1 mV/s; (b) Cycling performance at 5 mA/g and the first-cycle voltage profile (inset); (c) Comparison of the volumetric capacity and average discharge voltage; (d) Voltage profiles of the 2nd, 5th, 10th, 20th, 30th, 40th, and 50th cycles at 5 mA/g; (e) Cycling performance of WSe₂ anode at 20 mA/g and the voltage profile at the 100th cycle (inset); (f) Cycling performance of the WSe₂ anode at 50 and 100 mA/g. Inset depicts the voltage profiles at the 100th cycle; (g, h) Rate capability and corresponding voltage profiles

We further conducted ex-situ XRD to investigate the underlying reaction mechanism. As shown in Fig. 3, the diffraction peaks at 13.6°, 31.4°, 37.8°, and 43.76° at the initial state (A point labeled in Fig. 3) correspond to (002), (100), (103), and (105) reflections of WSe₂, while the peaks at 38.47°, 44.78°, and 65.13° are the diffractions from the Al current collector (JCPDS no. 04-0850). Upon discharging to 0.5 V (B point), the diffraction peaks located at 13.6°, 31.4°, 37.8°, and 43.76° shift to 13.36°, 31.24°, 37.67°, and 41.5°, respectively. These peak shifts indicate the intercalation of K⁺ ions into the interlayer of WSe₂ and an increase of interlayer spacing. Upon further discharging to 0.1 V (C point), the correlative characteristic peaks of WSe₂ disappear completely. Meanwhile, new peaks appear at 15.35°, 20.06°, 21.23°, 27.30°, and 28.46° and can be assigned to the (002),

(211), (300), (322) and (132) lattice planes of K₂Se₄ (ICSD-430521). These observations indicate conversion-type reactions occurring during this step (from B to C). On reverse charging to 2 V (D point), the diffraction peaks associated with WSe₂ do not appear again, while new peaks at 23.52°, 24.23°, 31.01°, 34.02°, and 34.81° correspond well to the formation of Se (JCPDS no. 24-0714). According to these observations, the entire electrochemical reactions can be described as the following equations.



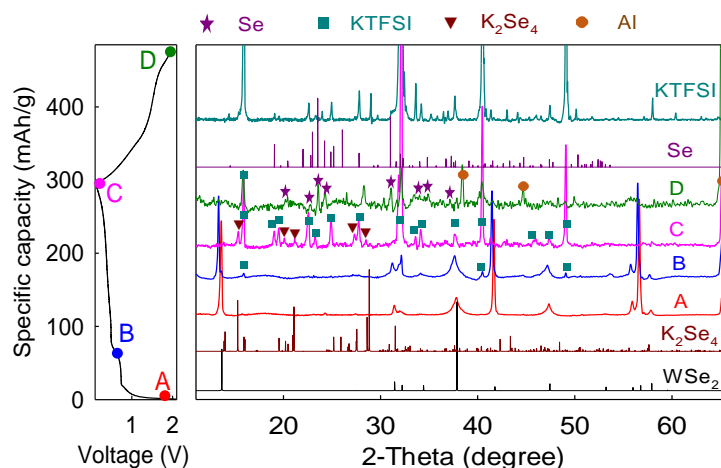


Fig. 3. Ex-situ XRD patterns of the WSe₂ electrode during the first cycle

It is noted that reactions (1) and (2) are irreversible, and after initial activation, the anode undergoes reversible conversion reactions between K₂Se₄ and Se, while the W metal formed during the initial cycle serves as an electron conductor to facilitate the conversion reactions.

We further fabricated proof-of-concept full-cell KIBs composed of an activated Prussian Blue cathode K_{1.89}Mn[Fe(CN)₆]_{0.92}^[35] and an activated WSe₂ anode with a

cathode-to-anode mass ratio of 1.8:1 (Fig. 4a). As depicted in Fig. 4b, the full-cell batteries operate at an average discharge voltage of 2.5 V and deliver a discharge capacity of 54.1 mAh/g and an energy density of 135.2 Wh/kg based on the total mass of the cathode and anode materials. After five cycles, the capacity and energy density remain at 45.8 mAh/g and 114.5 Wh/kg, respectively. This full-cell performance demonstrates the application prospect of the WSe₂ anode.

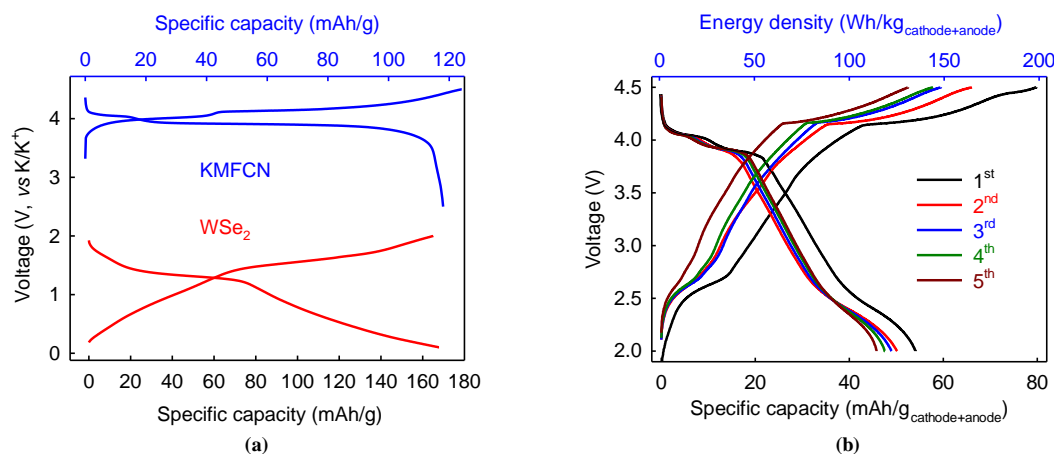


Fig. 4. (a) Voltage profiles of K_{1.89}Mn[Fe(CN)₆]_{0.92} cathode and WSe₂ anode at 10 mA/g; (b) Electrochemical performance of the full-cell battery

4 CONCLUSION

In summary, we have revealed the intrinsic electrochemical behaviors of WSe₂. As an anode, WSe₂ exhibits an ultrahigh volumetric capacity of 1772.8 Ah/L at a current density of 5 mA/g, good rate capability (72 mAh/g at 200 mA/g), and long-term cyclability (83.14% capacity retention over 100 cycles at 100 mA/g). Through ex-situ XRD analysis we revealed that WSe₂ first undergoes

intercalation reactions and subsequently conversion reactions to form K₂Se₄ and W during initial activation, and follows reversible conversion reactions between K₂Se₄ and Se in the subsequent cycles. Furthermore, full-cell KIBs utilizing the WSe₂ anode and a Prussian Blue cathode can provide an average discharge voltage of 2.5 V and an energy density of 135.2 Wh/kg_{cathode+anode}. WSe₂ may open new opportunities for the exploration of high-volumetric-capacity anode material in rechargeable potassium-ion batteries.

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