2D black TiO$_{2-x}$ nanoplate-decorated Ti$_3$C$_2$ MXene hybrids for ultrafast and elevated stable lithium storage

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**ABSTRACT**

Novel 2D black TiO$_{2-x}$ nanoplate-decorated Ti$_3$C$_2$ MXene (TiO$_{2-x}$/Ti$_3$C$_2$) hybrids were rationally designed and prepared by a facile wet process. The MXene nanosheets, serving as the carriers, not only enable the rapid electron and ion transport at the interface, but also avoid the aggregation of TiO$_{2-x}$ nanoplate during lithiation and de-lithiation. On the other hand, the presence of TiO$_{2-x}$ nanoplate also acts as the spacer to avert the MXene nanosheets from severe restacking. The successful formation of TiO$_{2-x}$/Ti$_3$C$_2$ were examined by X-ray diffraction (XRD) analysis, fourier transform infrared spectroscopy (FT-IR), scanning electron spectroscopy (SEM), transmission electron spectroscopy (TEM) and X-ray photoelectron spectroscopy (XPS). When served as anodes in lithium ion batteries (LIBs), the resulting TiO$_{2-x}$/Ti$_3$C$_2$ hybrid electrode delivers high specific capacity of 131 mA h g$^{-1}$ even cycling at a high current density of 5 A g$^{-1}$ (17 C) for 500 times and superior rate capability (even at a current density of 34 C, the specific capacity still remains at 115 mA h g$^{-1}$), which highlight its great promise in the fields of energy storage and conversion.

1. Introduction

Lithium ion batteries (LIBs) represent a cutting-edge energy storage technology for storing variable energies such as solar and wind energy, and are efficient and sentient next-generation power sources for a wide range of applications due to the unique advantages, such as low self-discharge rate, high energy density, no memory effect and long cycle life [1–4]. However, it should be pointed out that the electrochemical performance of LIBs is usually restricted by the intrinsic properties of electrodes.

In the past decade, compared to commercial graphite anodes, some representative anodic materials with high specific capacities such as Si, Ge and Sn have attracted much attention [5–7]. However, these materials suffer from a huge volume change during lithiation/de-lithiation process. This severe volume variation will lead to the pulverization of the electrode and unstability of the formed SEI film, thus will impede the cycle stability [8,9]. Recently, transition metal oxides (TMOs) have been in the spotlight of research effort because of their high considerable capacity, abundant resource, as well as low cost [10–12]. Among them, Titanium oxide (TiO$_2$) is considered as a promising candidate for alternative electrode material owing to the advantages in terms of safety, low-cost, and environmental friendly [13,14]. For example, Yu et al. [15] developed a feasible templating method to fabricate porous rutile TiO$_2$ submicroboxes. On account of the porous thin shells, exceptionally high surface area, and embedded small primary nanoparticles, the obtained TiO$_2$ submicroboxes exhibit significantly improved lithium storage properties with negligible capacity degradation for more than 500 cycles. However, it is undeniable that pure TiO$_2$ anodes still experience low specific capacity (~335 mA h g$^{-1}$) and poor rate performance. Therefore, it is still desirable to improve the
It is first reported in 2011 that black titania oxide (TiO$_{2-x}$) has been produced after reducing white TiO$_2$ using hydrogen at 200 °C under 20 bar pressure for 5 days [17]. TiO$_{2-x}$ exhibits high photolytic activity under solar light irradiation and improved lithium storage capability due to the abundant oxygen vacancy-contained characteristics [18–21]. Because of the presence of oxygen vacancies, TiO$_{2-x}$ shows better electrical conductivity and higher lithium storage capacity than that of white TiO$_2$, which were also have been confirmed by our previous study [22]. However, for practical applications, such as electrical vehicles, the rate performance of TiO$_{2-x}$ anodes are still not unsatisfactory and urgent to be enhanced. Recently, among the abundant two-dimensional materials, a new family of 2D materials called “MXene”, comprising few atoms thick layers of transition metal carbides, nitrides, or carbonitrides, endow great promise in many applications [23]. Usually, MXene features with consummate conjunction of graphene oxide and graphene characters when compared with graphene [24,25]. MXene also possesses attractive advantages such as excellent mechanical flexibility and rapid lithium transport, which make them ideal 2D candidates for energy storage [26,27].

In this work, based on our previous research, we further report facile synthesis of 2D TiO$_{2-x}$ nanoplate-decorated Ti$_3$C$_2$ MXene by solution method at room temperature. Herein, Ti$_3$C$_2$ MXene sheets act as excellent conductive two-dimensional support to load the black TiO$_{2-x}$ nanoplate. At this point, the MXene carrier not only enables rapid reversible lithium ions and electrons transport at the interface, but also prevent the TiO$_{2-x}$ aggregation during lithium ion insertion and extraction. In addition, the MXene carrier not only enables rapid reversible lithium ions and electrons transport at the interface, but also prevent the TiO$_{2-x}$ aggregation during lithium ion insertion and extraction. On the other hand, the interposed TiO$_{2-x}$ nanoplates also serve as the spacer to hold back MXene nanosheets restacking, thus preserving the active areas from being lost. When served as anodes in LIBs, the obtained TiO$_2$-x/Ti$_3$C$_2$ nanohybrids exhibit unprecedented rate performance and cycle stability. The specific capacity of TiO$_2$-x/ Ti$_3$C$_2$ nanohybrids anode reaches as high as 135 mA h g$^{-1}$, even cycled at high current density of 5 Ag$^{-1}$ (17C) for 500 times. Even the charging-discharging process is performed at current density of 10 A g$^{-1}$ (34 C), the specific capacity can still be maintained at around 110 mA h g$^{-1}$. This work is desirable expected to cater for a solution to foster the applications of TiO$_2$-x based anodes with ultra-high rate performance.

2. Experimental

2.1. Materials

Ti$_3$AlC$_2$ was purchased from Forsman Scientific (Beijing) Co., Ltd., China. Other chemicals and reagents were of analytical purity and used without further purification. Distilled water was used throughout.

2.2. Sample synthesis

2.2.1. Preparation of 2D TiO$_{2-x}$ (0 < x < 2) nanoplate

According to our previous work [22], in a typical synthesis, adding dropwise 6 ml of hydrofluoric acid (HF) into a flask containing 50 ml of titanium butoxide (TBOT) under stirring. Then, the mixture was poured into a Teflon-line stainless steel autoclave, the autoclave was where placed in a drying oven and kept at 190 °C for 24 h. After cooling, the formed jelly-like white precipitates were collected via centrifugation, washed and dried for further usage. In attempt to obtain black TiO$_2$-x nanoplate, a facile reduction process was carried out. Namely, 0.6 g of TiO$_2$ nanoplate and 0.6 g of sodium borohydride (NaBH$_4$) were grinded and mixed. Next, the mixture was loaded in a tube furnace with horizontal placement and annealing at 400 °C for 1 h under flowing nitrogen. The formed black product was washed with 1 M HCl solution and distilled water. Finally, black TiO$_2$-x nanoplate could be obtained after vacuum drying.

2.2.2. Preparation of Ti$_3$C$_2$ MXene nanosheets

For obtaining Ti$_3$C$_2$ MXene nanosheets, typically, 5 g of Ti$_3$AlC$_2$ was added to 50 ml of 40% HF solution and stirred at room temperature for three days, then centrifuged, washed several times with deionized water, and dried under vacuum at 60 °C overnight.

2.2.3. Synthesis of TiO$_{2-x}$/Ti$_3$C$_2$ nanohybrids

In a typical synthesis, 0.4 g of Ti$_3$C$_2$ nanosheets were added to 20 ml DMSO and stirred at room temperature for 18 h, then centrifuged, washed several times with deionized water. After that, Ti$_3$C$_2$ were dispersed into 200 ml of deionized water under nitrogen atmosphere, and ultrasonic dispersion for 1 h. On the other hand, 0.4 g of TiO$_{2-x}$ were dispersed into 50 ml of deionized water and ultrasonic dispersion for 20 min. Finally, mixing the dispersed Ti$_3$C$_2$ solution and TiO$_{2-x}$ dispersion and further stirred at inert atmosphere overnight. After that, the products were filtered and washed several times with deionized water, and dried under vacuum at 50 °C overnight.

2.3. Materials characterization

Powder X-ray diffraction (PXRD) data were collected on a Bruker D8 Advance instrument using a Cu Ka radiation (λ = 1.54056 Å) at room temperature. EPR analysis was conducted on Bruker EMX-10/12 apparatus. SEM images and Energy Dispersive Spectroscopy (EDS) were obtained on a Nova NanoSEM 450 field-emission scanning electron microscope at an acceleration voltage of 10 kV and 20 kV, respectively. Transmission electron microscopy (TEM) characterization was carried out using a JEM-2100F (Japan). Atomic force microscopy (AFM) was performed using a Si CANTILEVER with contact mode. X-ray photoelectron spectroscopy (XPS) measurements were recorded with an ESCALAB 250Xi. Nitrogen sorption isotherms were collected at 77 K (Micrometrics ASAP 2020 analyzer) after vacuum degassing of the sample at 120 °C for 10 h.

2.4. Electrochemical measurements

CR2025 cells were utilized as standard battery pack and assembled in an argon-filled glove box to evaluate electrochemical properties. TiO$_2$-x/Ti$_3$C$_2$ electrode was constructed with active material, conductive graphite, and polyvinylidene fluoride (PVDF) binder, the mass ratio was 8:1:1. Firstly, the three components were mixed and stirred in N-Methyl pyrrolidone (NMP) solvent and blade-coated on a piece of Cu foil. The foil was cut into disks of 14 mm in diameter after vacuum drying at 110 °C overnight. The electrolyte consisted of a solution of 1 M LiPF$_6$ in a mixture of 1:1 (vol/vol) dimethyl carbonate (DMC)/ ethylene (EC) (Shenzhen Kejingstar Technology Ltd., China). The counter electrodes were pure Li foils. The battery measurements were performed on a Neware battery testing device (Shenzhen, China) at the constant current mode over the range of 0.01–3 V. Cyclic voltammogram (CV) curves were obtained with voltage window ranging from 0.01 to 3 V using a CHI660D electrochemical workstation (Chenhua, Shanghai, China). The specific capacities were calculated based on the total weight of TiO$_{2-x}$/Ti$_3$C$_2$ hybrids. The theoretical capacity of TiO$_2$-x and Ti$_3$C$_2$ was considered as 335 and 320 mA h g$^{-1}$, respectively. For each electrode, the loading amount of active materials is around 1 mg cm$^{-2}$.

3. Results and discussions

The schematic for the preparation of 2D TiO$_{2-x}$/Ti$_3$C$_2$ nanohybrids is depicted in Scheme 1. Briefly, Ti$_3$C$_2$ MXene is fabricated by goal-oriented etching Al layers of the precursor (MAX, Ti$_3$AlC$_2$) using HF and finally delaminates into few or multi-layered MXene sheets. Subsequently, PXRD patterns SEM images of 2D black TiO$_{2-x}$ nanoplate and the white TiO$_2$ precursors were shown in Fig. 1. Fig. 1a shows the PXRD patterns of white TiO$_2$ and black TiO$_{2-x}$, respectively. It is revealed that the black TiO$_{2-x}$ still basically maintain the anatase phase structure after
NaBH₄ reduction, and the morphology was still unchanged, as shown in Fig. 1b and c. Moreover, the further AFM characterization shows that the thickness of black TiO₂₋ₓ nanoplate was nearly 3 nm were decorated on the surface and the interspaces of Ti₃C₂ MXene to form TiO₂₋ₓ/Ti₃C₂ nanohybrids under ambient conditions.

XRD analysis associated with Ti₃AlC₂, Ti₃C₂, and TiO₂₋ₓ/Ti₃C₂ were carried out to investigate the crystallographic phase of the products, which are shown in Fig. 2a. After HF treatment, the most intense XRD peak of Ti₃AlC₂ (2θ ≈ 38°, black curve in Fig. 2a) disappears. The (0 0 2) peak of Ti₃AlC₂ at 9.5° is shifted to lower 8.8° and is broadened in both Ti₃C₂ (red curve) and TiO₂₋ₓ/Ti₃C₂ nanohybrids (blue curve), implying the interlayer spacings were substantial expanded, as well as meaning that Al layers have been successfully removed of by etching and formation of Ti₃C₂ MXene. In addition, it is worth considering that species like atoms or molecules with unpaired electrons play an important role in some applications, which can qualitatively and quantitatively measured by EPR. In this regard, EPR measurements were carried out under ambient condition at 110 K to verify the presence oxygen vacancies. As shown in Fig. 2b, the pink line for the white TiO₂ nanoplate shows a negligible signal peak, while the black TiO₂₋ₓ nanoplate exhibits a very strong EPR signal centered on the magnetic field strength of 3478 G, which can be attributed to the unpaired electrons trapped on the surface oxygen vacancies [28,29].

The general morphology and microstructures of the Ti₃C₂ and TiO₂₋ₓ/Ti₃C₂ hybrids were characterized by SEM, TEM and HRTEM. Fig. 3a and d show the SEM images of Ti₃C₂ and TiO₂₋ₓ/Ti₃C₂, respectively. It can be clearly seen from Fig. 3a that the interlayers of bulk Ti₃AlC₂ material (Fig. 4a) are propped open after HF treatment, and the surface...
Fig. 2. (a) PXRD patterns of Ti₃AlC₂ (black), Ti₃C₂ (red), and TiO₂₋ₓ/Ti₃C₂ (blue); (b) EPR spectrums of white TiO₂ and black TiO₂₋ₓ.

Fig. 3. (a, d) SEM images of Ti₃C₂ and TiO₂₋ₓ/Ti₃C₂ nanohybrids, insert images in Fig. 3a and d show the corresponding magnified image, respectively; (b, e) TEM images of Ti₃C₂ and TiO₂₋ₓ/Ti₃C₂ nanohybrids; (c, f) HRTEM images of Ti₃C₂ and TiO₂₋ₓ/Ti₃C₂ composite; (g-j) EELS maps of (h) C, (i) O, and (j) Ti elements.

Fig. 4. (a) SEM image of Ti₃AlC₂ and (b) Typical nitrogen adsorption isotherms of TiO₂₋ₓ/Ti₃C₂ nanohybrids.
of the obtained Ti3C2 is smooth, which can also be confirmed by TEM (Fig. 3b). After formation of TiO2-x/Ti3C2 nanohybrids, as shown in Fig. 3d and e, the TiO2-x nanoplates are distributed on the surface and interspaces of Ti3C2 multilayers (the circle location in Fig. 3e). Moreover, Fig. 3c and f exhibit the HRTEM images of Ti3C2 and TiO2-x/Ti3C2, further confirming the production of pure Ti3C2 and TiO2-x/Ti3C2 nanohybrids. The clear lattice fringes with fringes spacing of 0.26 nm and 0.36 nm can be indexed as the Ti3C2 and anatase TiO2-x, respectively. The insert fast-fourier transform (FFT) patterns in Fig. 3c and f also confirm the successful preparation of Ti3C2 and TiO2-x/Ti3C2 nanohybrids.

Fig. 3g-j show the corresponding element mapping images of the TiO2-x/Ti3C2 nanohybrids, the relatively less amount C and O elements (Fig. 3h and i) are mainly attributed to the uneven distribution of TiO2-x within Ti3C2 multilayered nanosheets. At the same time, it should be noted that after intercalation, the TiO2-x/Ti3C2 nanohybrids get high specific surface area of 32.1 m^2 g^−1, as shown in Fig. 4b.

The chemical composition and chemical states of Ti, C and O elements in TiO2-x/Ti3C2 nanohybrids were investigated using X-ray photoelectron spectroscopy (XPS) technique. Fig. 5a illustrates the survey scan of the TiO2-x/Ti3C2 hybrids, the chemical compositions of Ti, C, O and F were detected. Among them, the only impurity F element can be attributed to the residue F ions from the HF solution [30]. In the Ti 2p XPS spectra, as shown in Fig. 5b, the Ti 2p XPS spectra of TiO2-x/Ti3C2 hybrids can be deconvoluted into four peaks. The two peaks located at binding energies of 459.3 eV (Ti 2p3/2) and 465.2 eV (Ti 2p1/2) are attributed to the lattice Ti-O bond in TiO2-x [31]. While the other two peaks at binding energies of 455.6 eV (Ti 2p3/2) and 460.7 eV (Ti 2p1/2) correspond to the lattice of Ti-C bond [32,33]. Furthermore, for C 1s XPS spectrum, three peaks at 281.9 eV, 285.1 eV and 286.8 eV can be observed in Fig. 5c after peak fitting, which can be assigned to the C-Ti, C=C and C-O, respectively [34]. Besides, the O 1s XPS spectrum (Fig. 5d) can be split into two peaks centered at 530.6 eV and 532.5 eV which belong to Ti-O-Ti and Ti-O-H bonds, respectively [35].

The electrochemical performances of TiO2-x/Ti3C2 anodic LIBs were investigated by using a 2025 type coin cell assembly with pure lithium foil as the counter electrode. Fig. 6a shows the cyclic voltammetry (CV) test results of TiO2-x/Ti3C2 nano-hybrid electrode for first three cycles with a voltage window of 0–3 V at a scan rate of 0.2 mV s^−1 vs. Li/Li^+. During the first lithiation step, two evident oxidation peaks which located at 1.72 and 0.63 V, as well as a weak broad peak at 2.37 V can also be detected, which can be assigned to the lithiation of TiO2-x and Ti3C2, respectively [36]. It should be considered that, in the subsequent cycles, the peaks at 1.72 and 0.63 V are disappeared, which is mainly ascribed to the formation of solid electrolyte interphase (SEI) film and the trapped Li^+ within the MXene layers [37]. It is a remarkable fact that there is slight shift and decrement regarding these peaks, implying a two-phase reaction mechanism during lithiation and de-lithiation processes based on the following equations [25].

\[
\begin{align*}
\text{Ti}_3\text{C}_2 + x\text{Li}^+ + xe^- & \rightarrow \text{Ti}_3\text{C}_2\text{Li}_x \quad (1) \\
\text{TiO}_{2-x} + x\text{Li}^+ + xe^- & \rightarrow \text{Li}_x\text{TiO}_{2-x} \quad (2)
\end{align*}
\]

Moreover, during the anodic process, reductive peaks at 1.07 and 2.55, as well as 2.05 V can be attributed to the de-lithiation of TiO2-x and TiO2-x/Ti3C2 nano-hybrids. Consider that after the first scan, the reductive peaks at 1.07 and 2.05 V remain nearly unchanged in terms of the intensity and position, proving the stability and fairly high electrochemical activity of TiO2-x/Ti3C2 nano-hybrid electrode. Fig. 6b exhibits the typical first three cycle discharge-charge voltage profiles of the TiO2-x/Ti3C2 electrode. The first discharge and charge specific capacities of TiO2-x/Ti3C2 electrode are 287 and 176 mA h g^−1, respectively, resulting in a corresponding coulombic efficiency of 61.3%. However, in the next two cycles, the coulombic efficiency goes up to more than 90%. The resulting low first cycle coulombic efficiency was mainly ascribed to the formation of SEI film and the irreversible reactions between the electrode and the electrolyte. At the same time, the apparent voltage plateaus between in 1.5–2 V in both charge and discharge curves are attributed to the de-lithiation and lithiation of TiO2-x/Ti3C2 hybrids, the phenomenon is completely consistent with the result of CV examination.

The rate capability of TiO2-x/Ti3C2 was further explored at increasing current densities, the test results were illustrated in Fig. 6c. The electrode afforded specific capacities of 206, 187, 168, 152, 131, and 115 mA h g^−1 at current densities of 0.1, 0.5, 1, 2, 5, and 10 A g^−1.
respectively. The electrode showed excellent reversibility with the specific discharge capacity recovered to the starting value when the current density returned to 0.1 A g\(^{-1}\) after cycling at high current densities. It is worth considering that based on the materials’ theoretical specific capacities and the actual active material loading amount on the electrode, the current density of 10 A g\(^{-1}\) corresponds to 34 C. Thus, even working at high current density of 34 C, the electrode still maintains a high specific capacity of 115 mA h g\(^{-1}\), which surpassing most of the reported works, as shown in Table 1. For comparison, we also fabricate bare TiO\(_{2-x}\) anodic LIBs. It can be clearly seen from Fig. 6f that, when cycling at the same current density of 5 C, the bare TiO\(_{2-x}\) electrode exhibits sharply decreased capacity retention of merely 25 mA h g\(^{-1}\). However, TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode still delivers a high specific capacity of around 170 mA h g\(^{-1}\). On the other hand, thus strongly demonstrating that after incorporation with Ti3C2, the electrical conductivity of TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode has been significantly improved, which in-turn enhancing the rate performance.

The long-term cycling stability of the TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode was evaluated by cycling at a large current density of 5 A g\(^{-1}\) (17 C). Before the long-term cycling, the electrode was firstly activated at a small current density. From Fig. 6d, it can be seen that in the initial cycles, the specific capacity of the TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode was sharply decreased due to the electrode activation process. After the electrode goes stability, the electrode exhibits a significantly stable specific capacity of around 130 mA h g\(^{-1}\) even working at high current density of 5 A g\(^{-1}\) (17 C) for 500 cycles.

Moreover, electrochemical impedance spectroscopy (EIS) was performed to further verify that the TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) nano-hybrid perform well in LIBs. The EIS spectrums of Ti3C2, TiO\(_{2-x}\) and TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrodes are shown in Fig. 6e. It is clearly seen from Fig. 6e that a semicircle in high-frequency region is formed and in the low-frequency region the spectra is linear. It is well known that during the discharge process, in some instances, both of the charge transfer reaction between the electrodes and the formed SEI film lead to the semicircle in the high-frequency region. Besides, the linear behavior in the low-frequency region is ascribed to the diffusion of Li ions on the electrodes [62,63]. From Fig. 6d, it is clearly shown that TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode shows a much smaller semicircular diameter among three electrodes, implying the best charge transfer efficiency. The result was ascribed to the better conductivity of TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) which possibly related to the formation of heterojunctions between TiO\(_{2-x}\) and Ti3C2 after hybridization [64], and this corresponded to the excellent rate performance of TiO\(_{2-x}/\text{Ti}_3\text{C}_2\) electrode.
In conclusion, the nano-hybrids of 2D TiO$_2$-x nanoplate-decorated multi-layered Ti$_3$C$_2$ MXene are prepared by a step-by-step assembly solution method. The synergistic effect of the presence of Ti$_3$C$_2$ MXene and TiO$_2$-x nanoplates are beneficial for the hybrid material fabrication and the improved battery performance. This novel material design efficiently combines the high electrical conductivity of Ti$_3$C$_2$ MXene and improved capacity of oxygen vacancy-contained TiO$_2$-x nanoplates. As a result, the TiO$_2$-x/Ti$_3$C$_2$ hybrid anodes exhibit excellent cyclability and rate capability, which may offer a new concept for developing ultra-stable and fast anodes for next-generation LIBs.

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