Two-Dimensional Vanadium Carbide (V_2C) MXene as Electrode for Supercapacitors with Aqueous Electrolytes

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Abstract

Recently, a large family of 2D materials called MXenes have attracted much attention in the field of supercapacitors, thanks to the excellent performance demonstrated by Ti_3C_2 MXene-based electrodes. However, research on MXenes for supercapacitor applications has been primarily focused on Ti_3C_2, even though there are more than 20 other members of this large family of materials already available. Studies on other MXenes are emerging, with promising results already achieved by Ti_2C, Mo_2C, and $Mo_{1.33}C$ in aqueous electrolytes. Yet, many other MXenes remain unexplored in aqueous supercapacitor applications. In this work, we report on the electrochemical behavior of a vanadium carbide MXene, V_2C, in three aqueous electrolytes. Excellent specific capacitances were achieved, specifically 487 F/g in 1 M H_2SO_4, 225 F/g in 1 M $MgSO_4$, and 184 F/g in 1 M KOH, which are higher than previously reported values for few micrometer-thick delaminated MXene electrodes. This work shows the promise of V_2C MXene for energy storage using aqueous electrolytes.

1. Introduction

The need to develop robust electrochemical energy storage solutions is driven by the rise of renewable energy sources (wind, solar, etc.) and the growing demand for high-performance rechargeable portable electronic devices (laptops, cell phones, cars, etc.)[1-2]. Supercapacitors store energy through two fast charge storage mechanisms: the double layer capacitance, which is the electrostatic separation of charges at the electrode/electrolyte interface when a potential is applied, and the pseudocapacitance, which originates from the fast surface redox reactions with a linear dependence of the charge stored on the potential window[3-6].

In recent years, a variety of new nanomaterials belonging to the family of MXenes has been discovered and explored for energy storage applications[7-8]. MXenes are transition metal carbide and/or nitride nanosheets, conferring good conductivity with tunable transition metal oxide-like surface termination that can undergo redox reactions. For example, the first discovered MXene, Ti_3C_2, has been investigated as an electrode for supercapacitors in a wide variety of electrolytes and its performance was shown to greatly depend on the electrolyte used. In neutral and basic electrolytes, capacitances between 45 F/g and 135 F/g were obtained and the charge storage mechanism was demonstrated to be pseudo-intercalation by *in-situ* X-ray diffraction[9]. Higher capacitances, up to 325 F/g, were obtained in sulfuric acid electrolyte thanks to the contribution of the hydrogen storage mechanism[10-12]. Recently, the gravimetric capacitance of Ti_3C_2-based electrode was further increased to 450 F/g using a thinner electrode (90 nm thick) in 3M H_2SO_4[13].

Other MXenes have rarely been investigated for aqueous supercapacitor applications. Due to its close chemical similarity with Ti_3C_2, Ti_2C was also tested in 1M KOH and showed capacitance up to 51 F/g[14]. Then, freestanding films of Mo_2C and $Mo_{1.33}C$ have been tested in 1M H_2SO_4 and showed capacitance of 196 F/g and 339 F/g, respectively[15-16]. From about 30 different synthesized MXenes, titanium and molybdenum carbides and their composites have been the only ones investigated. It is highly possible that other MXenes may exhibit better performance.

Among the large family of MXenes, V_2C is of particular interest as it is one of the lightest and the vanadium surface layers could potentially enable pseudocapacitive behavior. It was previously demonstrated that V_2C has an ion intercalation mechanism when this material was proposed as an electrode for lithium and sodium ion batteries[17-20]. Recently, V_2C has also been reported as transparent conductive thin films[21]. In this paper, we prepared a freestanding V_2C film and, for the first time, investigated its electrochemical behavior as an electrode for supercapacitors in three different aqueous electrolytes (H_2SO_4, KOH, and $MgSO_4$). These electrolytes were selected to gain insight on the effect of the electrolytes, because it was shown that Ti_3C_2 performance greatly depended on the electrolyte used. These three electrolytes were selected because of their clear difference, as one is acidic with the smallest cation H^+, one is neutral with divalent Mg^{2+}, and the last one is basic with a larger K^+ ion. The best performance was obtained in 1 M H_2SO_4 with a gravimetric capacitance up to 487 F/g at 2 mV/s.

2. Experimental Section

Commercial powders of vanadium (99.9% metals basis, 100–200 mesh), aluminum (99.8% purity, 300 mesh), and carbon black (99% purity, -300 mesh) were used as starting materials for the synthesis of V_2AlC. The molar ratio of V:Al:C was 2:1.1:0.9. The powders were ground with a mortar for 15 min then heated to 1550 °C under argon for 2 h in a tube furnace with a heating rate of 3 °C/min, and finally cooled down to room temperature. The resulting powder was treated in 100 mL of a 10 M H_3PO_4 solution at 80 ˚C for 15 h to dissolve and remove impurities from V_2AlC, such as Al_8V_5 and V_2C.

To prepare V_2C, 1 g V_2AlC powder (200 mesh) was added to 20 mL 28 M HF (49% HF, Aladdin) under stirring at room temperature for 45 h. This acidic mixture was diluted in de-ionized water (DI H_2O) followed by multiple centrifugation (8000 rpm, 6 min per cycle). After each centrifugation cycle, the supernatant was decanted into waste and the sediment was dispersed in DI H_2O until neutral pH (~ 6) is reached. The resulting product was then dried at 60 ˚C in a vacuum oven. The dried powder was collected using vacuum assisted filtration using PVDF membrane and wash copiously with more than 2000 mL of DI H_2O. The resulting product was then dried at 60 ˚C in a vacuum oven overnight.

Similar to delamination protocol for Ti_3C_2 powder,[22] V_2C powder (0.5 g) was delaminated by stirring in 20 mL of DI H_2O containing 3 mL of organic base tetramethylammonium hydroxide (TMAOH 25% in H_2O, Aladdin, China) for 12 h at room temperature. The TMAOH excess was then separated from the product by repeated centrifugation at 5000 rpm. The resulting wet sediment formed a clay-like paste that could be rolled between water-permeable membranes to produce flexible, free-standing films.

V_2C films were punched into 6 mm-diameter disks with mass loadings of 1.9 mg/cm^2 and used directly as binder-free electrodes. The electrochemical performance was investigated using a VMP3 potentiostat (Biologic, S.A.) with three-electrode Swagelok cells, where delaminated V_2C film was the working electrode, overcapacitive activated carbon was the counter electrode, cellulose membrane was the separator, and either a Hg/$HgSO_4$ electrode or an Ag wire was used as the reference electrode. 1 M H_2SO_4, 1 M KOH, and 1 M $MgSO_4$ solutions were used as electrolytes. The capacitance was calculated by integration of current with respect to time, according to the following equation:

$$C = \left(\int_0^{\frac{V}{s}} i dt\right)/(sVm)$$

Where C is the gravimetric capacitance (F/g), i is the gravimetric current density (A), s is the scan rate (V/s), and V is the potential window (V).

X-ray diffraction (XRD) patterns were collected using a Bruker D8 diffractometer operated at 40 KV and 40 mA using a Cu Kα radiation (λ = 1.5406 Å). Scanning electron microscopy (SEM, JSM-6700F) were used to observe the morphology of the delaminated V_2C film.

3. Results and discussion

Figure 1a shows a schematic of synthesis and delamination of V_2C. After selectively etching the Al-layer in V_2AlC using HF solution, the resulting multilayered V_2C powder was delaminated using TMAOH. After exposure to HF, the (002) peak in the XRD pattern of V_2AlC shifted to 8.6° (Figure 1b) corresponding to a *c*-lattice parameter of 20.5 Å and confirming synthesis of V_2C, while some unreacted V_2AlC remained. [18] Because the layers in the multilayered V_2C powder are bound to each other by weak van-der-Waals or hydrogen bonds, intercalation of large molecules such as TMAOH can weaken these bonds, leading to expansion and delamination of V_2C layers. The intercalated V_2C powder behaved like clay and could be rolled into a freestanding film. As a result, the c-lattice parameter of V_2C was further expended to 24.2 Å after intercalation and peaks corresponding to its (004) (006) and (008) are visible, suggesting that V_2C layers were reorganized during the rolling and drying steps. Although intercalation of TMAOH into Ti_3C_2 was shown to result in oxygenated and fluorinated surfaces,[23] the effects of surface termination were not studied in this work.

A cross-section scanning electron microscope (SEM) image of the rolled V_2C film shows that the film is composed of restacked nanosheets (Figure 1c). The rolled V_2C film exhibits good flexibility, as demonstrated in Figure 1d. The conductivity of transparent V_2C film was previously reported to be 3300 S/cm[21]. These suitable electronic and mechanical properties suggest that V_2C could be promising for flexible and wearable electronic devices.

The electrochemical behavior of this delaminated V_2C film was investigated in three aqueous electrolytes: 1 M H_2SO_4, 1 M KOH, and 1 M $MgSO_4$. Figure 2a, b and c show the corresponding cyclic voltammograms and Figure 2d summarize the specific capacitance obtained. The cyclic voltammograms obtained in all electrolytes are overall rectangular, indicative of capacitive behavior, but some broad peaks are noticeable in each case, suggesting pseudocapacitive redox processes[24]. Upon cycling in 1 M H_2SO_4 at 2 mV/s, there are clearly two reduction peaks, at -0.85 V and -1.00 V *vs.* Hg/$HgSO_4$, and two oxidation peaks, at -0.62 V and -0.95 V *vs.* Hg/$HgSO_4$, which should correspond to two redox couples. Upon cycling in 1 M KOH at 2 mV/s there is a less pronounced broad reduction peak at -1 V *vs.* Ag and a broad oxidation peak at -0.8 V *vs.* Ag. Upon cycling in 1 M $MgSO_4$ there are broad oxidation and reduction peaks around -0.7 V *vs.* Ag. The presence of these peaks implies possible electrochemical intercalation of cations, however further work is needed to clearly understand the origin of these redox peaks.

In terms of performance, the specific capacitances of V_2C were high in these three electrolytes. The best performance was obtained in 1 M H_2SO_4 with a capacitance up to 487 F/g at 2 mV/s. It is important to note that this is a higher capacitance compared to similar few μm-thick delaminated Ti_3C_2[10-11], $Mo_{1.33}C$[16], and Mo_2C[15] MXene electrodes cycled in 1 M H_2SO_4, which might be thanks to the lower molar mass of V_2C. At a higher rate of 100 mV/s, the capacitance decreased to 170 F/g demonstrating a reasonable rate capability. The restacked nanosheet morphology, obtained during freestanding film preparation, might decrease ion accessibility. This phenomenon was reported with Ti_3C_2 and its rate capability could be significantly improved by inserting nanotubes[25], or by forming vertically aligned electrodes[26]. The maximum capacitances obtained in 1 M KOH and 1 M $MgSO_4$ are respectively 184 F/g and 225 F/g. Compared to similar delaminated Ti_3C_2 electrode cycled in 1 M KOH and 1 M $MgSO_4$[9], V_2C is superior. At a higher scan rate of 100 mV/s, V_2C performances in 1 M KOH and 1 M $MgSO_4$ were close to each other, with the capacitances decreasing to 68 F/g and 57 F/g, respectively. These differences in specific capacitance among the three electrolytes might be due to their conductivity[27-28] and charge of the cation. Other factors, such as the ion size, solvation shell and energy of desolvation, cannot be excluded as well. Finally, it can be observed that the charge and discharge capacitance are not perfectly equal. Further work, such as *in-situ* XRD, is needed to clarify the reasons. A possible explanation is that during reduction (discharge) some cations are irreversibly intercalated and trapped between V_2C layers. Dissolution of vanadium during cycling is also possible, especially in acidic electrolyte[29].

To further investigate the potential of V_2C for supercapacitors applications, the cycle life of the V_2C was tested by galvanostatic charge-discharge at a high current density of 10 A/g. Figure 2e shows the relationship between the specific capacitance and the cycle number. The capacitance slightly decreased with the cycle number in H_2SO_4 but were stable in KOH and $MgSO_4$. More specifically, the capacitance retention after 10,000 cycles in 1 M H_2SO_4, 1M KOH and 1 M $MgSO_4$ were 83%, 94% and 99%, respectively. Pseudocapacitive materials, such as vanadium oxides[30], often suffer from shorter cycle life than carbon-based electrode, which rely only on double-layer mechanism. Thus, the capacitance losses observed in H_2SO_4 and KOH can be attributed to partially irreversible redox reactions, which might potentially lead to oxidation and degradation of V_2C considering its layers are thinner compared to Ti_3C_2. Also, vanadium from the carbide may be solving in acidic environment[29], as suggested by the presence of vanadium cations in the electrolyte after 10000 cycles in H_2SO_4, detected by inductively coupled plasma spectrometry. Figures 2f shows the charge and discharge curves obtained in H_2SO_4. Absence of steps during charge-discharge cycles exhibits a supercapacitor-like behavior.

4. Conclusions

This work shows that the electrochemical behavior of V_2C supercapacitor electrodes is dependent on the aqueous electrolyte composition. The maximum specific capacitance values obtained in 1 M H_2SO_4, 1 M KOH, and 1 M $MgSO_4$ were 487 F/g, 184 F/g and 225 F/g, respectively. Noteworthy, these results obtained from similar few micrometer-thick delaminated

MXene freestanding electrode are among the highest reported. These results are encouraging and suggest a potential for further exploration of vanadium carbide MXenes in energy storage applications. The electrochemical performance could further be improved via previously reported modification of Ti_3C_2, such as the preparation of hydrogel[13], layers vertical alignment[26], or hybridization with redox nanoparticles[31]. The full understanding of the pseudocapacitive charge storage mechanism of V_2C will require additional work to understand the origin of the redox reactions involved[12, 32-33], which could inspire other ways to improve the performance. Finally, since V_2C electrodes previously showed good performances upon non-aqueous lithium and sodium ion intercalation[17-20], their behavior in organic electrolytes should be studied[34-35].

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Figure 1. (a) Schematic representation of synthesis and intercalation-assisted delamination of V_2C. (b) Powder XRD patterns of V_2AlC precursor before (black line) and after (blue line) exposure to HF as well as of the rolled V_2C film (red line). (c) A cross-section SEM image of the rolled V_2C film. (d) An optical image of flexible V_2C film.

Figure 2. Electrochemical performance of V_2C electrode. Cyclic voltammograms (CVs) at different scan rates in (a) 1 M H_2SO_4, (b) 1 M KOH and (c) 1 M $MgSO_4$. (d) Specific capacitance measured from corresponding CVs. (e) Cycle lives measured from galvanostatic charge - discharge at 10 A/g and (f) profile in 1 M H_2SO_4.