



# Review A Review of Nb<sub>2</sub>CT<sub>x</sub> MXene: Synthesis, Properties and Applications

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Abstract:  $Nb_2CT_x$  is an important member of MXene family. It has attracted widespread attention because of its abundant functional groups, high hydrophilicity, high electrical conductivity as well as low ion transport barrier, showing great potential in various applications. In order to utilize the advantages of Nb<sub>2</sub>CT<sub>x</sub> MXene, the progress of preparation, properties and applications are reviewed in this work. This work focuses on different methods of  $Nb_2CT_x$  preparation and applications in electrochemical energy storage (supercapacitors and secondary batteries), electrocatalytic hydrogen evolution, photocatalytic hydrogen evolution, sensors, etc. Additionally, the main problems of self-stacking and prospect of Nb<sub>2</sub>CT<sub>x</sub> MXene are discussed.

Keywords: Nb<sub>2</sub>CT<sub>x</sub>; synthesis; application; energy conversion; electrochemical energy storage

# 1. Introduction

MXene, a 2D layer-structured material, has high conductivity, excellent mechanical property and large surface area [1,2]. MXene consists of transition metal nitrides, carbides or carbonitrides. Its molecular formula is  $M_{n+1}X_nT_x$ , where M is the transition metals, including Ti, Sc, Cr, V, Zr, Nb, and Mo, X is carbon or nitrogen and T<sub>x</sub> stands for surface terminals, including -F, -OH as well as -O groups [3,4]. Usually, MXene can be obtained by selectively etching the A atom (Si or Al atom) layers of MAX phase (precursor of MXene, M represents transition metal element; A represents main group element; X represents carbon or nitrogen.) [5,6]. Because of its chemical composition and unique structure, MXene and its composites have been applicated in electrochemical energy storage (supercapacitors and secondary batteries) [7], catalysis [8], sensors [9], electromagnetic interference shielding [10], etc.

So far, most of the research on MXene has focused on  $Ti_3C_2T_x$ , for the preparation of  $Ti_3C_2T_x$  is quite mature. In MXene materials, as the number of atomic layers increases, the molecular mass increases and the theoretical capacity decreases. Therefore, compared with  $Ti_3C_2T_x$ , MXene with  $M_2X$  structure has a higher specific capacity.  $Nb_2CT_x$  exhibits outstanding conductivity, near zero bandgap, and has special metallic properties because of the flexible combination of surface functional groups [11,12]. Nb<sub>2</sub>CT<sub>x</sub> can be obtained by acid etching of the Nb<sub>2</sub>AlC MAX phase. Theoretical studies have shown that Nb<sub>2</sub>CT<sub>x</sub> and its modified materials will show excellent performance in many fields because of the improved properties.

Figure 1 shows that  $Nb_2CT_x$  with different properties have been applied in various fields in recent years. Cheng et al. [13] have studied the charge storage mechanism of  $Nb_2CT_x$  in lithium-ion batteries, by combining density functional theory (DFT) calculations and complementary experiments. It is found that the outstanding electrochemical performance of  $Nb_2CT_x$  is mainly related to the unique structure of -O functional groups on Nb<sub>2</sub>CT<sub>x</sub> surface. As the oxidation states of O and Nb changes, Nb<sub>2</sub>CT<sub>x</sub> will store charge, while Li-ion can intercalate or deintercalation between the interlayers of  $Nb_2CT_x$  for charge compensation. The theoretical capacity value of  $Nb_2CT_x$  can reach 542 mAh/g [14]. Xin et al. [15] have shown that  $Nb_2CT_x$  nanosheet is an outstanding material as supercapacitor electrode by evaluating the theoretical quantum capacitances. It can be calculated



Citation: Guan, G.; Guo, F. A Review of Nb2CTx MXene: Synthesis, Properties and Applications. Batteries 2023, 9, 235. https://doi.org/10.3390/ batteries9040235

Academic Editors: Sylvain Franger and Pascal Venet

Received: 11 January 2023 Revised: 31 March 2023 Accepted: 13 April 2023 Published: 19 April 2023



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using the equation,  $C_Q = e^2 D(-Ve)$ , where D(E) is the electronic densities of state (DOS), *E* is the energy with respect to the Fermi level, *V* is the voltage of the electrode, and *e* is the elementary charge. The theoretical quantum capacitances of Nb<sub>2</sub>CT<sub>x</sub> electrode are 1091.1 and 1828.4 F/g at negative and positive electrodes, respectively. Nb<sub>2</sub>CT<sub>x</sub> is an excellent candidate for catalysis. In photocatalytic application, the structure of 2D Nb<sub>2</sub>CT<sub>x</sub> can promote photogenerated carrier to transfer from interior to Nb<sub>2</sub>CT<sub>x</sub> surface. This leads to improved separation efficiency and photodegradation performance [16]. Similarly, Nb<sub>2</sub>CT<sub>x</sub> has also been applied in electrocatalytic hydrogen evolution (HER) due to its abundant hydrophilic functional groups, especially the terminated O atoms. The Gibbs free energy ( $\Delta G^a_{H^*}$ ) for the adsorption of atomic hydrogen is close to the ideal value (0 eV) [17]. In addition, Nb<sub>2</sub>CT<sub>x</sub> has also been applied in sensors due to its highly hydrophilic surface and high charge-carrier mobility [18].



Figure 1. Different properties and applications of Nb<sub>2</sub>CT<sub>x</sub>.

The outstanding properties of Nb<sub>2</sub>CT<sub>x</sub> have ensured its applications in different areas, such as energy storage devices [19–22], electrocatalysis [23,24], photocatalysts [25,26], and sensors [27–29]. In addition, applications of Nb<sub>2</sub>CT<sub>x</sub> on other areas have also been reported, such as on perovskite solar cells (PVSCs) [30], photodetector [31], biomedicine [32], electromagnetic shielding [33], adsorbents [34], etc. In this paper, the progress of Nb<sub>2</sub>CT<sub>x</sub> preparation, its properties and different applications are reviewed, while also focusing on the exfoliating methods as well as applications in energy storage (supercapacitors and secondary batteries), photocatalytic hydrogen evolution, electrocatalytic hydrogen evolution, sensors, etc. Additionally, the main problems of self-stacking of Nb<sub>2</sub>CT<sub>x</sub> are discussed and prospects for the future are explored.

#### 2. Synthesis

In Nb<sub>2</sub>AlC MAX, the Nb-C bonds are strong, while the metal bonds of Nb-Al are relatively weak, therefore, Nb<sub>2</sub>CT<sub>x</sub> is usually obtained by selectively removing Al atom layers of Nb<sub>2</sub>AlC. Even though the metal bond energy between the Nb-Al atomic layers is relatively weak, the bonding strength is still greater than that of the Van der Waals force. Hence, it seems difficult to peel the precursor into few-layer or monolayer using ultrasound-assisted liquid-phase exfoliation or other mechanical stripping method. Chemical exfoliation method is the most effective and widely used approach, where Nb<sub>2</sub>CT<sub>x</sub> can be prepared by etching away the Al atomic layer without destroying the Nb-C atomic layers. Figure 2 shows the structures of various types of MAX phases and their corresponding MXene structures obtained using chemical exfoliation method.



**Figure 2.** Structures of MAX phases and their corresponding MXene. Reprinted with permission. Copyright John Wiley & Sons, Inc., 2013 [35].

Studies have also shown that  $Nb_2CT_x$  can be intercalated by a variety of metal cations [36] or organic molecules [20] to increase the interlayer spacing, and to facilitate the etching of Al atoms and exfoliation of  $Nb_2CT_x$  nanosheets. After exfoliation, the single-layer or few-layer  $Nb_2CT_x$  nanosheets can be obtained.

#### 2.1. Synthesis of Nb<sub>2</sub>AlC

Most of Nb<sub>2</sub>CT<sub>x</sub> is obtained from Nb<sub>2</sub>AlC MAX. The quality of Nb<sub>2</sub>AlC MAX affects the quality of Nb<sub>2</sub>CT<sub>x</sub> nanosheets directly. In order to get Nb<sub>2</sub>CT<sub>x</sub> nanosheets with high quality, Nb<sub>2</sub>AlC MAX with high quality should be prepared first. Many methods are developed to prepare Nb<sub>2</sub>AlC MAX, such as high-temperature solid-state reaction method [37–40], self-propagating high-temperature synthesis (SHS) process [41–44] and spark plasma sintering technique (SPS) [45].

Stumpf et al. [38] have synthesized Nb<sub>2</sub>AlC powders using the high-temperature solid-state reaction method. Firstly, high-purity Nb, NbC and Al powders are uniaxially compacted at 250 MPa to obtain powder particles, then Nb<sub>2</sub>AlC powder is obtained by grounding particles at a high temperature of 1600 °C. Yeh et al. [41] have synthesized Nb<sub>2</sub>AlC using a productive self-propagating SHS process. To obtain Nb<sub>2</sub>AlC, Al<sub>4</sub>C<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub> and Al undergo chemical reactions under different stoichiometric parameters and high temperatures (1200–1800 °C). Although these methods can prepare high-purity Nb<sub>2</sub>AlC, the equipment and high temperature required for the synthesis lead to huge investment and production costs.

SPS is considered as an effective method for the synthesis of many ceramics. Compared with the thermocompression method, SPS shows obvious advantage in heating as it can be heated by pulsed direct current, and requires lower temperature and shorter cycle time. Zhou et al. [45] have synthesized Nb<sub>2</sub>AlC using spark SPS, whereinhigh-purity Nb, Al and NbC powders are uniaxially compacted at 20 MPa and then vacuum sintered in a spark plasma at a heating temperature of 1450 °C.

Among all these preparation methods mentioned above, the preparation of Nb<sub>2</sub>AlC using the SPS method has advantages in temperature, reaction time and cost, thus promises to be an efficient and commonly used method for the synthesis of Nb<sub>2</sub>AlC.

# 2.2. Synthesis of $Nb_2CT_x$

 $Nb_2CT_x$  is usually obtained from  $Nb_2AIC$  MAX using the chemical exfoliation methods. Commonly used chemical etching methods include hydrofluoric acid (HF) etching [46–50], hydrofluoric acid/lithium fluoride (HCl/LiF) in situ generation of hydrofluoric acid etching [51,52], hydrothermal etching [53], Lewis acid molten salt etching [54,55] and electrochemical etching [56]. Etching with a high-concentration HF solution is currently the most common method for preparing Nb<sub>2</sub>CT<sub>x</sub>. Using the HF etching method, Zhao et al. [46] have obtained few-layer Nb<sub>2</sub>CT<sub>x</sub> nanosheets by changing the reaction temperature, time and other parameters, shown in Figure 3a. After HF etching, the densely packed layers of Nb<sub>2</sub>AlC become unconsolidated, and the angle of (002) diffraction peak is smaller due to action of different surface functional groups and interlayer water molecules. In the meantime, Nb<sub>2</sub>CT<sub>x</sub>, exposed to the solution will combine with different functional groups (-OH, -F and -O). However, some Nb<sub>2</sub>AlC usually remains in the Nb<sub>2</sub>AlC prepared using the etching method. In Table 1, different experimental parameters of HF etching method is compared, since etching conditions will affect the purity and layer spacing of Nb<sub>2</sub>CT<sub>x</sub> [14,46,57–61]. As shown in Table 1, proper acid concentration, reaction temperature and reaction time will promote the etching reaction and benefit in obtaining pure Nb<sub>2</sub>CT<sub>x</sub> nanosheets.



**Figure 3.** Synthesis and delamination of Nb<sub>2</sub>CT<sub>x</sub>. (**a**) Synthesis of Nb<sub>2</sub>CT<sub>x</sub> using HF etching method. Reprinted with permission. Copyright Royal Society of Chemistry, 2019 [46]. (**b**) Synthesis of Nb<sub>2</sub>CT<sub>x</sub> using HCl/LiF in situ generation of hydrofluoric acid etching method. Reprinted with permission. Copyright Elsevier, 2021 [51]. (**c**) Synthesis of Nb<sub>2</sub>CT<sub>x</sub> using electrochemical etching method. Reprinted with permission. Copyright John Wiley & Sons, Inc., 2020 [56]. (**d**) Delamination of Nb<sub>2</sub>CT<sub>x</sub> using isopropylamine (i-PrA). Reprinted with permission. Copyright John Wiley & Sons, Inc., 2015 [20]. (**e**) Delamination of Nb<sub>2</sub>CT<sub>x</sub> by intercalation with tetramethylammonium hydroxide (TMAOH). Reprinted with permission. Copyright Elsevier, 2020 [16].

Etching Solution	Temperature/°C	Time/h	References
50% HF	25	240	[14]
40% HF	60	90	[46]
48% HF	55	48	[57]
50% HF	55	72	[58]
48% HF	80	24	[59]
50% HF	30	24	[60]
40% HF	50	72	[61]

**Table 1.** Different parameters of HF etching method for the preparation of  $Nb_2CT_x$ .

Since the high concentration of hydrofluoric acid is harmful to humans and the environment, developing environmentally friendly and milder methods to prepare  $Nb_2CT_x$  is urgent and important. Hydrofluoric acid/lithium fluoride (HCl/LiF) in situ generation of hydrofluoric acid etching method is an effective alternative to the HF etching method. As Figure 3b shows, Xiao et al. [51] have obtained few-layer Nb<sub>2</sub>CT<sub>x</sub> using HCl/LiF in situ generation of hydrofluoric acid etching method, where  $Li^+$  can be embedded in the Nb<sub>2</sub>CT<sub>x</sub> layers to facilitate the delamination of Nb<sub>2</sub>CT<sub>x</sub>nanosheets. However, HF is still generated and released during the HCl/LiF in situ generation of hydrofluoric acid etching process, and thus this method is harmful to humans and environment in some ways. Therefore, it is imperative to propose green preparation methods which can avoid the direct use and release of hydrofluoric acid. Peng et al. [53] have obtained  $Nb_2CT_x$  using solvothermal treatment by mixing hydrochloric acid (HCl) and sodium tetrafluoroborate (NaBF<sub>4</sub>) together, followed by ultrasonication and intercalation. Specifically, the mixture of NaBF<sub>4</sub>, HCl and Nb<sub>2</sub>AlC is hydrothermally reacted at 180 °C for 15–35 h. The etching process takes place in a confined hydrothermal system, without exposure to toxic hydrofluoric acid vapors. The hydrothermal method can remove the Al layer more completely compared to traditional HF etching method.

Since -F functional groups are electronegative, too much -F functional groups will greatly reduce the conductivity of MXene [36], so proposing fluorine-free etching methods is also necessary. In recent years, some fluorine-free etching methods have been reported, such as to obtain  $Ti_2CT_x$  using electrochemical etching method [62] and to obtain  $Ti_3C_2T_x$  by etching  $Ti_3AlC_2$  using the molten salt method [63]. Similar to these methods of obtaining  $Ti_3C_2T_x$  and  $Ti_2CT_x$ , fluorine-free methods to obtain  $Nb_2CT_x$  have also been reported in recent years.

Song et al. [56] have synthesized fluoride-free Nb<sub>2</sub>CT<sub>x</sub> flakes using an electrochemical etching method (E-etching method), as shown in Figure 3c. This method is carried out in a three-electrode system where Nb<sub>2</sub>AlC phase powder and carbon black are mixed and evenly drop-cast onto the CFC substrate. This electrode is oxidized in HCl electrolyte for 4 h at 50 °C with a voltage of 1 V to etch away the Al atomic layers. Li et al. [55] have used a fluoride-free method using direct redox coupling of the cation from the Lewis acid molten salt and the Al atom of Nb<sub>2</sub>AlC. Specifically, AgCl is used as salt, while Nb<sub>2</sub>AlC is mixed with AgCl, KCl and NaCl in a molar ratio of 1:5:2:2, and reacted at 700 °C for about 24 h. During this reaction, Al atoms will be oxidized by AgCl and eventually removed from Nb<sub>2</sub>AlC to obtain Nb<sub>2</sub>CT<sub>x</sub>.

In general, hydrofluoric acid etching is the most commonly used method for synthesizing  $Nb_2CT_x$ . However, considering the danger of hydrofluoric acid to humans and the fact that fluorine functional groups will reduce the electrochemical performance of  $Nb_2CT_x$ , many fluorine-free etching methods have been suggested. However, these methods still have problems, such as harsh reaction conditions or low yields. Therefore, it is still important to explore more efficient fluorine-free etching methods.

#### 2.3. Delamination of $Nb_2CT_x$

After the Nb<sub>2</sub>AlC etching reaction, the Al atoms are selectively removed, and replaced with functional groups, such as -F, -OH as well as -O. The obtained Nb<sub>2</sub>CT<sub>x</sub> layers are held together by hydrogen bonds and weak Van der Waals forces. However, the Nb<sub>2</sub>CT<sub>x</sub> sheets are multi-layered, and further delamination/exfoliation processing is required to form single-layer/few-layer Nb<sub>2</sub>CT<sub>x</sub> sheets. The specific surface area of two-dimensional materials will be greatly increased after further delamination.

Generally, the materials for delamination involve organic, ionic molecules or inorganic cation. Non-alkaline organic solvent intercalation is an effective way to obtain fewlayer Nb<sub>2</sub>CT<sub>x</sub>. Such organic solvent intercalants include DMSO [26,64], isopropylamine (i-PrA) [20,48], etc. For example, Mashtalir et al. [20] have obtained few-layer Nb<sub>2</sub>CT<sub>x</sub> sheets using isopropylamine (i-PrA), as shown in Figure 3d. When mixed with water, i-PrA can form ammonium cation R-NH<sub>3</sub><sup>+</sup>, which can be intercalated between Nb<sub>2</sub>CT<sub>x</sub> layers using electrostatic force. In addition, the i-PrA molecule has a three-carbon-atom alkyl tail, which can overcome the intercalation steric hindrance and push the Nb<sub>2</sub>CT<sub>x</sub> nanosheets apart, resulting in weaker interlayer interactions and leading to the delamination of Nb<sub>2</sub>CT<sub>x</sub>.

Cation intercalation, treating Nb<sub>2</sub>CT<sub>x</sub> with alkalization, is another widely used method. The intercalants include LiOH [14], KOH [36], TMAOH [16,65], and TPAOH [28,66,67]. As shown in Figure 3e, Cui et al. [16] have obtained few-layer Nb<sub>2</sub>CT<sub>x</sub> sheets by intercalating with tetramethylammonium hydroxide (TMAOH) for about 3 days at room temperature. Thus, the numerous functional groups on the surface of Nb<sub>2</sub>CT<sub>x</sub> nanosheet make it negatively charged. In order to achieve charge balance, H<sup>+</sup> will enter the interlayers. When treated with alkali, the cations of the alkali will replace H<sup>+</sup> and insert between the layers. After cation intercalation, the layer spacing of Nb<sub>2</sub>CT<sub>x</sub> will be larger and the -F functional groups will be replaced by hydroxyl functional groups. The intercalated alkali cations will weaken the interaction between the Nb<sub>2</sub>CT<sub>x</sub> sheets, so single-layer/few-layer Nb<sub>2</sub>CT<sub>x</sub> flakes can be obtained after simple exfoliation, such as shaking or water-bath sonication.

# 3. Structure and Properties of Nb<sub>2</sub>CT<sub>x</sub>

# 3.1. Structure of $Nb_2CT_x$

The Scanning Electron Microscope (SEM) image (Figure 4a) and Transmission Electron Microscope (TEM) image (Figure 4b) show that the few-layer Nb<sub>2</sub>CT<sub>x</sub> sheets are ultrathin and transparent. The high-resolution transmission electron microscopy (HRTEM) image (Figure 4c) and the selected area electron diffraction (SAED) pattern image (inset of Figure 4c) of the Nb<sub>2</sub>CT<sub>x</sub> nanosheets show good crystallinity. A clear crystal lattice pattern of (0001) crystal plane can be seen, showing good single crystal characteristics [46]. The X-ray diffraction (XRD) image (Figure 4d) indicates that the (002) peak of few-layer Nb<sub>2</sub>CT<sub>x</sub> sheets shifts toward a lower angle, suggesting a larger interspacing due to the elimination of Al atoms [57].



**Figure 4.** Characterization and structure of Nb<sub>2</sub>CT<sub>x</sub>. (a) SEM image of Nb<sub>2</sub>CT<sub>x</sub>, (b) TEM image of Nb<sub>2</sub>CT<sub>x</sub>, (c) HRTEM image of Nb<sub>2</sub>CT<sub>x</sub>. Reprinted with permission. Copyright Royal Society of Chemistry, 2019 [46]. (d) XRD image of Nb<sub>2</sub>CT<sub>x</sub>. Reprinted with permission. Copyright American Chemical Society, 2022 [57]. (e) Structure of Nb<sub>2</sub>C and Nb<sub>2</sub>CX<sub>2</sub> [68]. Copyright John Wiley & Sons, Inc., 2015.

The crystal structure of computationally modeled Nb<sub>2</sub>C MXene is similar to that of 1T-MoS<sub>2</sub>, including three layers with Nb-C-Nb sequence in a  $3 \times 3 \times 1$  supercell. The C layer is sandwiched by two Nb layers; C layers and Nb layers are stacked along the hexagonal c-axis [57]. Because of the highly unsaturated surface in Nb<sub>2</sub>C, functionalized elements (F, O, OH and N) can be easily trapped in the hollow sites. Figure 4e [68] exhibits the modeling structures, including Nb<sub>2</sub>C and Nb<sub>2</sub>CX<sub>2</sub>, where X groups can be located at top of a Nb (1) atom (top layer), a C atom and a Nb (2) atom (bottom layer), and both Nb<sub>2</sub>C sides, therefore, there are a total of six configurations of adsorption sites.

# 3.2. Properties of $Nb_2CT_x$

Seredych et al. [69] have explored the thermal stability of Nb<sub>2</sub>CT<sub>x</sub> using mass spectrometry analyses and combined thermogravimetric. Nb<sub>2</sub>CT<sub>x</sub> etched by HF tends to release water at 246 °C and the main phase transition occurs at 807 °C, with a weight loss of 10–15 wt%. The weight loss coincides with the massive release of CO<sub>2</sub> as well as CO, indicating a possible transformation of Nb<sub>2</sub>CT<sub>x</sub> into the stable corresponding massive carbides. The Nb<sub>2</sub>CT<sub>x</sub>, further delaminated by TMAOH, has a weight loss of about 8.5 wt%, in a temperature range of 50 °C to 600 °C. NH<sub>3</sub>, NH<sub>2</sub>, and NH<sub>4</sub><sup>+</sup> species release because of the decomposition process and desorption process of TMA<sup>+</sup> ions. The results indicate that the intercalant is strongly bound to the surface of Nb<sub>2</sub>CT<sub>x</sub> nanosheets to hinder water adsorption. The transformation temperature slightly increases to 820 °C with less mass loss of about 4 wt%, indicating a higher stability of Nb<sub>2</sub>CT<sub>x</sub>. Therefore, intercalants, such as TMAOH, can replace water and affect the decomposition products at low-temperature, making Nb<sub>2</sub>CT<sub>x</sub> with better thermal stability.

Babar et al. [70] have explored the magnetic properties of Nb<sub>2</sub>CT<sub>x</sub>. The peculiar magnetic properties of Nb<sub>2</sub>CT<sub>x</sub> shows the existence of Meissner effect, and Nb<sub>2</sub>CT<sub>x</sub> is supposed to have superconductivity (SC) with the transition temperature of 12.5 K. Evaluated with the DFT first-principles calculation, the Nb<sub>2</sub>CT<sub>x</sub> shows a negative magnetic moment, which corresponds well to the experimental diamagnetic nature. Both the experimental analysis and calculation indicate Nb<sub>2</sub>CT<sub>x</sub> showing a superconductor-like diamagnetic behavior. The superconductivity-like magnetic property in Nb<sub>2</sub>CT<sub>x</sub> can be attributed to the higher density-of-states at the Fermi level and strong electron–phonon interaction [71].

Xu et al. [72] have studied the electronic properties and structural stability of Nb<sub>2</sub>CT<sub>x</sub> based on DFT calculations, paying special attention to the influence of surface groups. The study suggests that Te and O groups are more inclined to locate at the central C hollow sites on both Nb<sub>2</sub>CT<sub>x</sub> sides. The interaction strength between different functional groups and Nb atoms will gradually decrease from O to S, Se and to Te. This study has further analyzed bonding interactions using projected crystal orbital Hamiltonian populations. As the surface terminal gains less electrons from O to Te group, the integrated projected crystal orbital Hamilton population (pCOHP) value decreases gradually from O to Te. The altered interactions can also affect the electronic performance of Nb<sub>2</sub>CT<sub>x</sub>. This study has found that it is the different types of surface groups that determine the electronic properties and conductivity of Nb<sub>2</sub>CT<sub>x</sub>, with adsorption sites as a secondary factor. That is, Nb<sub>2</sub>CO<sub>2</sub> and Nb<sub>2</sub>CTe<sub>2</sub> are metallic, while Nb<sub>2</sub>CS<sub>2</sub> and Nb<sub>2</sub>CSe<sub>2</sub> can be metallic or semiconducting, depending on the adsorption sites of different surface groups on Nb<sub>2</sub>CT<sub>x</sub>.

Halim et al. [73] have obtained Nb<sub>2</sub>CT<sub>z</sub> and Ti<sub>2</sub>CT<sub>z</sub> film via physical vapor deposition on sapphire and LiF/HCl etching method, and then measured the optical and electronic properties of these films. Ti<sub>2</sub>CT<sub>z</sub> film exhibits metallic behavior while Nb<sub>2</sub>CT<sub>z</sub> film is not metallic, which can be supported by optical properties via spectroscopic ellipsometry measurements. The difference in optical response between Nb<sub>2</sub>CT<sub>z</sub> film and Ti<sub>2</sub>CT<sub>z</sub> film can be attributed to the absorption of Nb<sub>2</sub>CT<sub>z</sub> film near infrared. Additionally, the resistivity of Nb<sub>2</sub>CT<sub>z</sub> film increases when temperature decrease, because of the percolation between Nb<sub>2</sub>CT<sub>z</sub> flakes. The different optical and electronic properties of these films are influenced by the type of transition metal. Palisaitis et al. [74] have investigated the stability of Nb<sub>2</sub>CT<sub>x</sub> in plan-view geometry using electron energy loss spectroscopy (EELS), atomically resolved scanning transmission electron microscopy (STEM) as well as STEM simulations. This research has found that the Nb adatoms on Nb<sub>2</sub>CT<sub>x</sub> surface can attract and be linked to ambient O and then form clusters which will ripen over time, leading to the destruction of Nb<sub>2</sub>CT<sub>x</sub> structure stability. Considering the structure stability at a long time, the adatoms on the Nb<sub>2</sub>CT<sub>x</sub> surfaces are detrimental for Nb<sub>2</sub>CT<sub>x</sub> structure. Echols et al. [75] have explored ways to enhance the oxidation stability of Nb<sub>2</sub>CT<sub>x</sub>. It is found that the shelf life of Nb<sub>2</sub>CT<sub>x</sub> can be significantly improved with low temperature storage, using antioxidants, increasing the concentration of Nb<sub>2</sub>CT<sub>x</sub> solution, etc.

# 4. Applications

# 4.1. Secondary Batteries

MXene has rich chemical and structural diversity, and is a promising material for secondary batteries electrode. When the layer of MXene materials increases, the molecular mass increases, so the theoretical capacity will decrease [76]. Hence, compared to  $M_3X_2$  and  $M_4X_3$ , the  $M_2X$  structured MXene tends to have a higher specific capacity [77]. Nb<sub>2</sub>CT<sub>x</sub> MXene, showing low diffusion barriers, fast ion mobility and low average intercalation potentials, has generated great interests in recent years. Currently, the research on Nb<sub>2</sub>CT<sub>x</sub> application on secondary batteries is still at the exploratory stage. Taking lithium-ion batteries as an example, Nb<sub>2</sub>CT<sub>x</sub> has a low lithium-ion diffusion barrier (<0.1 eV), and thus Li<sup>+</sup> ions can easily intercalate into the Nb<sub>2</sub>CT<sub>x</sub> nanosheets. The surface functional groups of Nb<sub>2</sub>CT<sub>x</sub> with -O functional groups are higher than that of Nb<sub>2</sub>CT<sub>x</sub> with -F and -OH functional groups can inhibit the adsorption of lithium ions, which is detrimental to the electrochemical properties. The reaction of Nb<sub>2</sub>CT<sub>x</sub> with -O functional groups to adsorb lithium ions is as follows:

$$Nb_2CT_xO_2 + 2Li \leftrightarrow Nb_2CT_xO_2Li_2$$
 (1)

# 4.1.1. Lithium Ion Battery

As an important type of energy storage device, Lithium-ion batteries (LIBs) are playing an increasingly important role in our society owing to their ability to offer power for both our daily life and industrial manufacturing [78–80]. Numerous studies on Li-ion battery materials have been reported in recent years. For example, the first DFT calculations of a high-energy, 5 Volt rechargeable Li-ion battery cathode material Li<sub>2</sub>CoMn<sub>3</sub>O<sub>8</sub> were performed by Eglitis and Borstel [81]. It was found that the reversible capacity of Nb<sub>2</sub>CT<sub>x</sub> is higher than that of Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>, and the capacity value of Nb<sub>2</sub>CT<sub>x</sub> can reach 542 mAh/g [14], which is much larger than that of Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> (320 mAh/g). However, considering the strong Van der Waals force between Nb<sub>2</sub>CT<sub>x</sub> layers, Nb<sub>2</sub>CT<sub>x</sub> nanosheets are easy to aggregate or stack together. The self-restacking will lead to densely packed structure, reducing the specific surface area and increasing difficulty in the transportation of ions, thus to decreasing the rate performance and cycling performance of Nb<sub>2</sub>CT<sub>x</sub> after 200 cycles at 500mA is less than 200 mAh/g, which is far from the theoretical value. Therefore, strategies should be carried out to improve the electrochemical properties from Nb<sub>2</sub>CT<sub>x</sub>.

Reports have been published to solve the above problems in order to fully utilize the electrochemical properties of Nb<sub>2</sub>CT<sub>x</sub> [82–86] Li et al. [82] have mixed Nb<sub>2</sub>CT<sub>x</sub> with 1,3,5-benzenetricarboxylic acid (BA) to obtain BA-Nb<sub>2</sub>C, as shown in Figure 5a. BA molecules can be anchored into Nb<sub>2</sub>CT<sub>x</sub> nanosheets through hydrogen bonding interaction between Nb<sub>2</sub>CT<sub>x</sub> sheets and BA molecules, enlarging the interlayer spacing and enhancing the stability of Nb<sub>2</sub>CT<sub>x</sub>. Figure 5b shows that BA-Nb<sub>2</sub>C exhibits a larger specific surface area which can facilitate the Li<sup>+</sup> diffusion, provide more active sites, and improve the capability



rate of Nb<sub>2</sub>CT<sub>x</sub>. The BA-Nb<sub>2</sub>C electrode exhibits a high specific capacitance of 328.2 mAh/g at 0.1A/g, as shown in Figure 5c.

**Figure 5.** BA-Nb<sub>2</sub>C as anode materials for LIBs. (a) Schematic illustration of the synthesis mechanism, (b) Nitrogen adsorption–desorption isotherms of pure Nb<sub>2</sub>C and BA-Nb<sub>2</sub>C, (c) cycling stability of BA-Nb<sub>2</sub>C at 0.1 A/g. Copyright Springer Nature, 2022 [82].

The surface functional group of MXene plays an important role in the electrochemical reaction [84,85]. The -F functional and hydroxyl groups on Nb<sub>2</sub>CT<sub>x</sub> surface can hinder the diffusion of Li-ions in the interlayer, thereby greatly reduce the lithium storage specific capacity [86]. However, the -O groups on Nb<sub>2</sub>CT<sub>x</sub> surface will promote the diffusion and adsorption of Li-ions and effectively inhibit the formation of lithium dendrites [13]. Zhao et al. [14] have mixed LiOH with Nb<sub>2</sub>CT<sub>x</sub> to introduce more oxygen-containing functional groups and analyzed the corresponding electrochemical properties and Li storage dynamics. The surface functional groups (-O, -OH and -F) of Nb<sub>2</sub>CT<sub>x</sub> nanosheets changes when annealed at different temperatures. After annealing at 400 °C, -O surface functional groups of Li<sup>+</sup> intercalated Nb<sub>2</sub>CT<sub>x</sub> increase while the -OH and -F groups decrease. The Li-Nb<sub>2</sub>CT<sub>x</sub>-400 electrode shows a high specific capacity of 448 mAh/g at 0.05 A/g owing to the large number of -O surface functional groups.

### 4.1.2. Sodium Ion Battery

Sodium ion batteries (SIBs) have become a promising alternative to LIBs because of certain advantages, such as low cost, abundant sodium resources and similar energy storage mechanism. However, the radius of Na-ion is larger than that of Li-ion, and the development of SIBs is constrained by the short of suitable anode materials [87,88]. MXene, with its unique properties and structure, is promising to serve as a new type of anode material for SIBs [89,90]. As shown in Figure 6a, Yuan et al. [77] have constructed a robust 3D Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C composites, by hydrothermal synthesis of MoS<sub>2</sub> and carbon coating on Nb<sub>2</sub>CT<sub>x</sub> framework. The Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C composites show a reinforced cross-linked three-dimensional structure (Figure 6b). The high specific surface area and stable structure can better utilize the electrode material. The Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C electrode shows an improved charge transfer efficiency, and avoid structural collapse or volume expansion caused by Na-ion intercalation. The Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C exhibit a high capacity of 530 mAh/g after 200 cycles test in 0.1 A/g (Figure 6c). Zhang et al. [91] have prepared a sandwich-like composite including N-doped carbon nanotubes (N-CNTs) and Nb<sub>2</sub>CT<sub>x</sub> nanosheets using the electrostatic self-assembly approach. The N-CNTs are trapped between two MXene layers as conductive agents and spacers. In addition, the N-doped sites on the carbon nanotube walls can act as defects, promoting the diffusion and storage of Na-ion. The N-CNT@Nb<sub>2</sub>C is supposed to have good electrochemical performance as Na-ion batteries anode. Xiao et al. [92] have synthesized Ag-Nb<sub>2</sub>CT<sub>x</sub> composite by physically mixing Ag nanoparticles and Nb<sub>2</sub>CT<sub>x</sub> nanosheets. The Ag-Nb<sub>2</sub>CT<sub>x</sub> can provide larger specific surface area and higher conductivity. The Ag-Nb<sub>2</sub>CT<sub>x</sub> shows a capacity of 183 mAh/g at 0.05 A/g after 50 cycles in Na-ion battery.



**Figure 6.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in SIBs. (a) Schematic illustration of preparation of the Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C hybrid, (b) TEM image of Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C, (c) Cycling stability of Nb<sub>2</sub>CT<sub>x</sub>@MoS<sub>2</sub>@C at 1 A/g. Copyright American Chemical Society, 2021 [77].

### 4.1.3. Potassium Ion Battery

Potassium-ion batteries (KIBs) is also a potential alternative to LIBs because of the low cost and abundance of potassium-related resources. However, the ion migration of electrodes is difficult because of the much larger radius of K-ion compared to Li-ion [93,94]. Therefore, developing suitable electrode materials for KIBs is essential. The unique structure and excellent conductivity of Nb<sub>2</sub>CT<sub>x</sub> make it an ideal electrode candidate for KIBs [95]. As shown in Figure 7a, Liu et al. [58] have prepared in situ carbon-intercalated and surfacemodified Nb<sub>2</sub>CT<sub>x</sub> as electrode for Potassium-ion batteries. The carbon-intercalated Nb<sub>2</sub>C nanocomposite ( $Nb_2C/C$ ) can be obtained by intercalating dodecylamine (DDA) into the Nb<sub>2</sub>CT<sub>x</sub> layers, followed by annealing (Figure 7b). The Nb<sub>2</sub>C/C electrode shows less surface functional groups, enlarged interlayer spacing as well as increased specific surface area. The Nb<sub>2</sub>C/C electrode, with high electrical conductivity and good  $K^+$  diffusion coefficient, performs a specific capacity of 199.8 mAh/g at 0.02 A/g (Figure 7c). Mao et al. [20] have prepared self-standing composites of  $Nb_2CT_x$  and reduced graphene (rGO) (3D-rGO/Nb<sub>2</sub>C) using the electrostatic self-assembly approach. The paper electrode with 3D porous network can provide more active sites and promote the transfer of K<sup>+</sup>. The 3D-rGO/Nb<sub>2</sub>C shows a good capacity of 139 mAh/g after 1000 cycles at 500 mA/g, owing to the diffusion and pseudocapacitive mechanism in potassium ion storage.



**Figure 7.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in KIBs. (a) Schematic illustration for the synthesis of Nb<sub>2</sub>C/C, (b) HRTEM image of Nb<sub>2</sub>C/C, (c) Specific capacity of Nb<sub>2</sub>C/C at 0.02 A/g. Copyright Elsevier, 2022 [58].

### 4.1.4. Lithium Sulfur Battery

Lithium-sulfur (Li-S) batteries have been considered as the best alternative to LIBs because of advantages such as long cycle life, low cost of active materials, as well as high energy density. However, the commercialization of this technology has been plagued by some key challenges, such as the insulating shuttling effect of sulfur cathodes, volume changes during charging/discharging, and poor electronic/ionic conductivity [96,97]. As previously reported, MXene with unique two-dimensional structure, negatively charged surface, and high conductivity can immobilize soluble polysulfides by adsorption to suppress the shuttling effect [98,99]. As shown in Figure 8a, Song et al. [100] have designed Nb<sub>2</sub>C/Nb<sub>2</sub>O<sub>5</sub> heterostructures using water vapor etching. The Nb<sub>2</sub>C has high adsorption capacity, while  $Nb_2O_5$  can catalyze the activity of lithium polysulfides. Meanwhile, the accordion-like and porous structure (Figure 8b,c) formed during water vapor etching can suppresses the aggregation of Nb<sub>2</sub>C sheets and Nb<sub>2</sub>O<sub>5</sub> nanoparticles, promote Li<sup>+</sup> transport and facilitate diffusion of the anchored lithium polysulfides. The S-Nb<sub>2</sub>C/Nb<sub>2</sub>O<sub>5</sub> electrode exhibits a capacity of 621 mAh/g after 500 cycles test at 1.0 C (Figure 8d). By freeze-drying, Zhong et al. [101] have prepared petal-like Nb<sub>2</sub>CT<sub>x</sub> and MoS<sub>2</sub> is grown on the surface of  $Nb_2CT_x$  via hydrothermal method. The  $Nb_2CT_x$  and  $MoS_2$  is deemed to form a heterojunction owing to their high lattice adaptability. The  $MoS_2/Nb_2C$  heterojunction structure is favorable for the conductivity and adsorption capacity of lithium polysulfides. The specific capacity of  $MoS_2/Nb_2C$  cathode is 919.2 mAh/g at the current density of 0.2 C.

In addition to LIBs, SIBs, KIBs, and LSBs, Nb<sub>2</sub>CT<sub>x</sub> has also been applied to other batteries. The calcined Nb<sub>2</sub>CT<sub>x</sub> cathode used for aluminum batteries (Al batteries) shows a specific capacity of 08 mAh/g at 0.2 A/g after 500 cycle tests [57]. Li et al. [102] have proposed a new method of achieving high-energy-density MXene electrodes with a distinct discharge voltage plateau for aqueous zinc ion batteries. The authors use a high-voltage-scanning approach to trigger the behavior of Nb<sub>2</sub>CT<sub>x</sub> MXene cathode in aqueous Zn-ion batteries, which shows a stable Zn<sup>2+</sup> storage with a 1.55 V plateau. The energy density of the MXene electrode reaches 146.7 Wh/kg, which is much higher than that of the capacitive Nb<sub>2</sub>CT<sub>x</sub> electrode and other conventional cathodes.



**Figure 8.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in LSBs. (a) Schematics of preparation of S-Nb<sub>2</sub>C/Nb<sub>2</sub>O<sub>5</sub> heterostructures, (b,c) TEM images of Nb<sub>2</sub>C/Nb<sub>2</sub>O<sub>5</sub>, (d) Cycling stability of S-Nb<sub>2</sub>C/Nb<sub>2</sub>O<sub>5</sub> electrodes at 1.0 C. Copyright Elsevier, 2022 [100].

Table 2 lists the performance of  $Nb_2CT_x$  with its derivative materials for secondary batteries by category.

Battery	Materials	Current Density	Cycles	Capacity (mAh/g)	References
LIB	Li-Nb <sub>2</sub> CT <sub>x</sub> -400	2 A/g	2000	342	[14]
LIB	Nb <sub>2</sub> CT <sub>x</sub> -CNT	2.5 C	300	430	[20]
LIB	Chl@Nb <sub>2</sub> C	0.5 A/g	200	163	[77]
LIB	BA-Nb <sub>2</sub> C	$1 \mathrm{A/g}$	500	261.7	[82]
LIB	Nb <sub>4</sub> N <sub>5</sub> @Nb <sub>2</sub> C	2  A/g	800	109.2	[83]
KIB	3D-rGO/Nb2C	0.5 A/g	1000	139	[21]
KIB	Nb <sub>2</sub> C/ rGO	2  A/g	500	301.7	[94]
SIB	Nb <sub>2</sub> CT <sub>x</sub> @MoS <sub>2</sub> @C	$1 \mathrm{A/g}$	2000	403	[76]
SIB	Ag-Nb <sub>2</sub> CT <sub>x</sub>	0.05 A/g	50	183	[93]
Li-S	$Nb_2C/Nb_2O_5$	1.0 C	500	621	[100]
Li-S	$MoS_2/Nb_2C$	0.2 C	200	919.2	[101]
AIB	$Nb_2CT_x$	0.5 A/g	500	80	[57]

Table 2. The specific capacities of  $Nb_2CT_x$  for secondary batteries.

The restacking of Nb<sub>2</sub>CT<sub>x</sub> will impair the electrochemical properties, by hindering the transport of electrolyte ions inside the electrode and reducing the utilization of active sites in Nb<sub>2</sub>CT<sub>x</sub> electrode materials. the functional groups also greatly affect the electrochemical properties. In this section, different applications of Nb<sub>2</sub>CT<sub>x</sub> in secondary batteries have been introduced, to provide solutions or strategies in improving the electrochemical performance of Nb<sub>2</sub>CT<sub>x</sub>. Methods of intercalation in Nb<sub>2</sub>CT<sub>x</sub> nanosheets can effectively inhibit the restacking, with the intercalation materials of organic compounds [82,96], carbon materials [91], and metals [93]. Regulation the functional groups of Nb<sub>2</sub>CT<sub>x</sub> (for example, by substituting -O functional groups for -F, and -OH [14]), can obviously improve the ability of Nb<sub>2</sub>CT<sub>x</sub> to store charges. Studies have also shown that, increasing the ion transport channels of Nb<sub>2</sub>CT<sub>x</sub> by manufacturing porous or three-dimensional structures can also effectively improve the electrochemical performance [20,76,100]. All these methods have obvious effects on the specific capacity, cycle life or rate performance of  $Nb_2CT_x$ .

#### 4.2. Supercapacitors

Supercapacitors have been an important and widely applicated electrochemical energy storage device because of their high power density, fast charge–discharge capability and long-cycle stability [59,103]. For electrode materials of supercapacitors, the charge storage mechanism is an electrical double-layer capacitance mechanism and pseudo-capacitance mechanism [104,105]. In electric double-layer capacitive mechanism, ions store charges through electrostatic adsorption at the interface between supercapacitor electrodes and electrolytes, while in pseudocapacitive mechanism, ions store charges via fast reversible redox reactions on the surface of active materials. MXene with abundant surface functional groups, high electrical conductivity and rapid redox reactions on the surface is deemed to be an ideal pseudocapacitive material [106,107].

 $Nb_2CT_x$  is theoretically predicted to be an excellent electrode material with higher specific areal capacitance and gravimetric capacitance than  $Ti_3C_2T_x$ .  $Nb_2CT_x$  can provide redox-active Nb sites for pseudocapacitive-intercalation and the Nb element in  $Nb_2CT_x$  has a wider chemical valence or better electrochemical activity compared with that of Ti element [108]. However, the strong Van der Waals force between  $Nb_2CT_x$  sheets easily results in the aggregation of  $Nb_2CT_x$  nanosheets, leading to the degradation of electrochemical performance of  $Nb_2CT_x$  electrode. Recently, many works on  $Nb_2CT_x$  in supercapacitors have been devoted to improve the electrochemical performance. s. The electrochemical performance of  $Nb_2CT_x$  and its composites are listed in Table 3.

**Table 3.** The specific capacities of  $Nb_2CT_x$  for supercapacitors.

Materials	Electrolyte	Capacitance	References
Nb <sub>2</sub> C/Ti <sub>3</sub> C <sub>2</sub>	$1 \text{ M H}_2 \text{SO}_4$	584 F/g at 2 A/g	[59]
Nb <sub>2</sub> CT <sub>x</sub> /CNT	$1 \text{ M H}_2\text{SO}_4$	202 F/g at 2 mV/s	[109]
$Ti_3C_2T_x/Nb_2CT_x$	$2 \text{ M H}_2 \text{SO}_4$	370 F/g at 2 mV/s	[60]
$Co_3O_4$ -Nb <sub>2</sub> CT <sub>x</sub>	6 M KOH	1016 F/g at 2 mV/s	[110]
$Nb_2CT_x$	$1 \text{ M LiSO}_4$	154 F/g at 5 mV/s	[111]
S-P-NNO/f-Nb <sub>2</sub> CT <sub>x</sub>	1 M LiPF <sub>6</sub>	157 mAh/g at 2.0 A/g	[112]

Nasrin et al. [59] have prepared  $Nb_2C/Ti_3C_2$  (NCTC) heterointerface nanostructures via one-pot synergistic in situ etching of Nb<sub>2</sub>C/Ti<sub>3</sub>C<sub>2</sub>. The NCTC is synchronized to enhance surface dynamics of charge storage by exposing more active sites and ion diffusion channels. With the individual and synergistic charge storage at the heterointerface, the NCTC shows a pseudocapacitive property with a specific capacitance of 584 F/g at 2 A/g. Xiao et al. [109] have mixed carbon nanotubes with  $Nb_2CT_x$  to enlarge the interlayers spacing of Nb<sub>2</sub>CT<sub>x</sub> sheets. The conductive CNT can improve the rate ability of Nb<sub>2</sub>CT<sub>x</sub>/CNT. The Nb<sub>2</sub>CT<sub>x</sub> /CNT//AC asymmetric supercapacitor, with Nb<sub>2</sub>CT<sub>x</sub>/CNT and activated carbon as electrodes, exhibits an areal capacitance of  $462.0 \text{ mF/cm}^2$ . Li et al. [60] have prepared flexible all-MXene hybrid films by mixing  $Nb_2CT_x$  and  $Ti_3C_2T_x$  together. With the introduction of  $Ti_3C_2T_x$  nanosheets, the interlayer spacing of  $Nb_2CT_x$  is increased and the stacking of MXene sheets is hindered. The flexible all-MXene film supercapacitor demonstrates a specific capacitance of 370 F/g at 2 mV/s and a good performance rate. As shown in Figure 9a, Shen et al. [110] have introduced  $Co_3O_4$  into  $Nb_2CT_x$  layers using the electrostatic self-assembly approach to form a robust 2D cross-linked structure. Figure 9b shows that Co<sub>3</sub>O<sub>4</sub> nanoparticles (marked in yellow circles) are uniformly distributed on  $Nb_2CT_x$  surface to suppress the self-restacking of  $Nb_2CT_x$  sheets and provide more active sites. The 2D cross-linked Co-MXene electrode shows a specific capacitance of 730 F/g at 20 mV/s (Figure 9c). Li et al. [22] have constructed Nb<sub>2</sub>CT<sub>x</sub> hydrogels via a 4D printing using a heat-stimulated self-assembly process, with the conducting polymer PEDOT:PSS working as the crosslinker. The 3D porous structured  $Nb_2CT_x$  hydrogels, shows with



enhanced mechanical strengths, high specific surface area, good electrical conductivities and good electrochemical performance.

**Figure 9.** Applications of  $Nb_2CT_x$  in supercapacitors. (a) Schematic illustration of the preparation procedure of Co-MXene, (b) SEM images of the Co-MXene composites, (c) Specific capacitance of Co-MXene at different current densities. Copyright Elsevier, 2022 [110].

Liao et al. [111] have studied the capacitance behavior of Nb<sub>2</sub>CT<sub>x</sub> under cation intercalation by intercalating different cations. Compared to Na<sup>+</sup> and K<sup>+</sup>, the insertion of Li<sup>+</sup> tends to achieve better electrochemical performance and capacitance behavior. Nb<sub>2</sub>CT<sub>x</sub> electrode in the Li<sub>2</sub>SO<sub>4</sub> electrolyte shows a higher specific capacitance of 120 F/g at 20 mV/s, due to the fast transport and easy intercalation of the small-sized Li<sup>+</sup> between Nb<sub>2</sub>CT<sub>x</sub> layers.

Hybrid capacitors, with battery-type and capacitive-type materials as electrodes, can take into consideration high power density of supercapacitors as well as high energy density of batteries. Recently, the application of Nb<sub>2</sub>CT<sub>x</sub> in hybrid capacitors has attracted extensive attention. As shown in Figure 10a, Qin et al. [112] have prepared single-crystal perovskite NaNbO<sub>3</sub> nanocubes (S-P-NNO NCs) using a hydrothermal alkalization method with few-layer Nb<sub>2</sub>CT<sub>x</sub> (f-Nb<sub>2</sub>CT<sub>x</sub>). The f-Nb<sub>2</sub>CT<sub>x</sub> and S-P-NNO NCs are further assembled via freeze-drying to obtain S-P-NNO/f-Nb<sub>2</sub>CT<sub>x</sub> composites. Benefiting from the 3D-connected porous conductive network (Figure 10b) and synergistic effect between f-Nb<sub>2</sub>CT<sub>x</sub> and S-P-NNO NCs, the composites in Lithium-ion capacitors (LICs), shows a capacity of 157 mAh/g at 2.0 A/g (Figure 10c) and an energy density of 56 Wh/kg at the power density of 13 kW/kg in a full hybrid cell (Figure 10d).

At present, the experimental conditions of research on  $Nb_2CT_x$  are relatively mild and are mainly focused on aqueous electrolyte. Since the electrochemical stability of  $Nb_2CT_x$ MXene is relatively poor, designing a stable structure and optimizing application conditions to inhibit the oxidation and dissolution of  $Nb_2CT_x$  are also necessary.



**Figure 10.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in hybrid supercapacitors. (a) Schematic illustration of the synthetic process of the S-P-NNO/f-Nb<sub>2</sub>CT<sub>x</sub>, (b) TEM image of S-P-NNO/f-Nb<sub>2</sub>CT<sub>x</sub>, (c) Rate properties of S-P-NNO/f-Nb<sub>2</sub>CT<sub>x</sub> and other Nb-based oxide anodes, (d) Ragone plots of S-P-NNO/f-Nb<sub>2</sub>CT<sub>x</sub> for LICs. Copyright Royal Society of Chemistry, 2021 [112].

#### 4.3. Electrocatalytic Hydrogen Evolution

Electrocatalytic hydrogen evolution reaction (HER) is an important clean and efficient hydrogen production method, as hydrogen is considered to be an effective substitute for traditional fossil fuels [113,114]. Recent studies have proven that MXene-based catalysts have potential application in HER [115], or the -O functional groups on MXene surface are the active sites for HER [116]. Figure 11a provides a volcano map of the theoretical exchange current and the average Gibbs free energy of hydrogen adsorption. The predictions have shown that, the high binding strength of Nb<sub>2</sub>CO<sub>2</sub> and H leads to a slightly poorer HER catalytic performance than that of  $Ti_3C_2O_2$  [17]. Currently, some studies are devoted to weakening the hydrogen adsorption of Nb<sub>2</sub>CT<sub>x</sub> and improving the electrocatalytic hydrogen evolution activity.



**Figure 11.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in electrocatalytic hydrogen evolution reaction. (**a**) Volcano curve of exchange current (i<sub>0</sub>) as a function of the average Gibbs free energy of hydrogen adsorption ( $\Delta G^{a}_{H^*}$ ). Copyright American Chemical Society, 2016 [17]. (**b**) Schematic illustration of the preparation of Pt/Nb<sub>2</sub>CT<sub>x</sub>-600 catalyst, (**c**) TEM image of Pt/Nb<sub>2</sub>CT<sub>x</sub>-600, (**d**) Electrocatalytic HER performance of Pt/Nb<sub>2</sub>CT<sub>x</sub>-600 in 0.5 M H<sub>2</sub>SO<sub>4</sub>, (**e**) CA curves of Pt/Nb<sub>2</sub>CT<sub>x</sub>-600 and Pt/C catalysts for HER performance. Copyright MDPI, 2021 [116].

Anchoring single transition metal atom into 2D materials is an important strategy to develop electrocatalysts with high HER performance. As shown in Figure 11b, Fan et al. [117] have immobilized platinum nanoclusters on Nb<sub>2</sub>CT<sub>x</sub> via mechanochemical ball milling method to get electrocatalysts for hydrogen evolution. Ultrafine Pt<sub>3</sub>Nb nanoclusters are formed on the Pt/Nb<sub>2</sub>CT<sub>x</sub> composite after annealing at 600 °C (Figure 11c) and the average diameter of  $Pt_3Nb$  nanoclusters is 1.5 nm. The  $Pt/Nb_2CT_x$ -600 demonstrates a low overpotential of 46 mV and 5 mV at the current density of 100 mA/cm<sup>2</sup> and 10 mA/cm<sup>2</sup>, respectively (Figure 11d). After 20 h test, the  $Pt/Nb_2CT_x$ -600 also shows 5% degradation (Figure 11e). The good HER property can be attributed to more active sites and homogenous Pt dispersion after the thermal treatment and mechanochemical process. Zhang et al. [118] have studied the effect of surface functionalization of Nb<sub>2</sub>CT<sub>x</sub>-supported Pd catalysts for HER performance. Compared with oxygen-rich materials, the F-rich Nb<sub>2</sub>CT<sub>x</sub>-based Pd nanocatalyst shows better HER property and long-term stability. The Pd/Nb2C-HF shows an overpotential of 34 mV at 10 mA/cm<sup>2</sup>, with the Tafel slope of 34 mV/dec. The improved HER property of Pd/Nb<sub>2</sub>C with F-rich terminated catalyst can be attributed to the weakened adsorption of hydrogen, which make the evolution of hydrogen easier. Zhao et al. [23] have anchored Co single atoms on  $Nb_2CT_x$  to obtain Co@  $Nb_2CT_x$  composites. Because of the stronger hybridization between surface-terminated  $O_{2p}$  orbitals of  $Nb_2CT_x$  and  $Co_{3d}$ orbitals, the Co atoms in the Co@ Nb<sub>2</sub>CT<sub>x</sub> have an optimized electronic structure, leading a lower HER energy barriers and accelerated catalytic kinetics.

Except for anchoring transition metals, other strategies to prepare effective catalysts are also adopted. Jiang et al. [24] have tailored 2D Nb<sub>2</sub>CT<sub>x</sub> nanosheets into quantum dots (QDs), with active sites at the Nb<sub>2</sub>CT<sub>x</sub> QDs edges enriched. The intermediate H\* (absorbed hydrogen) species can be more moderately bound with metal atoms at the edge of Nb<sub>2</sub>CT<sub>x</sub> QDs, delivering a lower energy barrier. Nb<sub>2</sub>CT<sub>x</sub> QDs exhibits 6.8 times higher activity than pure Nb<sub>2</sub>CT<sub>x</sub> nanosheets and has good stability.

#### 4.4. Photocatalytic H<sub>2</sub> Evolution

 $Nb_2CT_x$  is an ideal candidate for photocatalyst because of its rich functional groups, hierarchical lamellar structure and good photocatalytic properties.

As shown in Figure 12a, Huang et al. [64] have prepared 2D CdS/2D Nb<sub>2</sub>CT<sub>x</sub> composite using the electrostatic self-assembly and solvothermal reaction. After the hydrothermal reaction of 2D CdS with  $Nb_2CT_x$ , the 2D CdS nanosheets will cover  $Nb_2CT_x$ , and form a flower-like morphology (Figure 12b). The photocatalytic  $H_2$  evolution rate of  $CdS/Nb_2CT_x$  composite is about 5040 µmol/g/h, which is 4.3 times higher than that of pure 2D CdS (Figure 12c). In CdS/Nb<sub>2</sub>CT<sub>x</sub> composite, Schottky heterojunction formed can the redistribution of charges on this 2D CdS/2D Nb<sub>2</sub>CT<sub>x</sub> composite and accelerate the photo-generated charge separation and transfer of 2D CdS nanosheets, thus to promote the water splitting reaction with more photo-generated electrons. Cui et al. [16] have synthesized ultrathin  $Bi_2WO_6/Nb_2CT_x$  composite photocatalyst using the hydrothermal method. The  $Nb_2CT_x$  flakes can greatly improve the separation efficiency of photogenerated carriers and boost the photocatalytic activity of Bi<sub>2</sub>WO<sub>6</sub>. Through in situ calcination, Makola et al. [119] have prepared a metal-free Nb<sub>2</sub>CT<sub>x</sub>@g-C<sub>3</sub>N<sub>4</sub> Schottky junction photocatalyst. The heterostructure exhibits a narrow energy bandgap and can significantly suppress electron recombination rate, indicating potential application in the photocatalytic. Peng et al. [25] have synthesized Nb<sub>2</sub>O<sub>5</sub> nanorod arrays on Nb<sub>2</sub>CT<sub>x</sub> nanosheets via hydrothermal method, followed by photodepositing Ag nanoparticles to form hierarchical composites. The Ag/Nb<sub>2</sub>O<sub>5</sub>@Nb<sub>2</sub>CT<sub>x</sub> nanohybrid exhibits 3.6 times higher photocatalytic  $H_2$  evolution rate than Nb<sub>2</sub>O<sub>5</sub> nanoparticles. The performance can be explained using the photogeneration efficient of electron-hole pairs provided by  $Nb_2O_5$  nanorods and abundant active sites provided by Ag nanoparticles. Tayyab et al. [120] have synthesized  $In_2S_3/Nb_2O_5/Nb_2C$  (INNC) via a one-pot in situ hydrothermal method. During the hydrothermal process,  $Nb_2CT_x$  flakes are partial oxidized into  $Nb_2O_5$  nanorods, and In<sub>2</sub>S<sub>3</sub> nanoparticles are chemically anchored on Nb<sub>2</sub>CT<sub>x</sub> flakes and Nb<sub>2</sub>O<sub>5</sub> nanorods. Due

to the heterojunction of  $In_2S_3$ / Nb<sub>2</sub>O<sub>5</sub>, INNC composite has an improved photoexcited charge carrier transfer with a higher photocatalytic H<sub>2</sub> evolution rate (68.8 µmol/g/h) than Nb<sub>2</sub>O<sub>5</sub>/Nb<sub>2</sub>C or In<sub>2</sub>S<sub>3</sub>. Xu et al. [121] have synthesized Ru/Nb<sub>2</sub>O<sub>5</sub>@Nb<sub>2</sub>C using one-pot method. During the hydrothermal reaction, Nb<sub>2</sub>C is partially oxidized into semiconductor Nb<sub>2</sub>O<sub>5</sub> nanowires and Ru<sup>3+</sup> is reduced to Ru on Nb<sub>2</sub>O<sub>5</sub>/Nb<sub>2</sub>C in the deposition process. The Ru/Nb<sub>2</sub>O<sub>5</sub>@Nb<sub>2</sub>C with metal/semiconductor/Nb<sub>2</sub>CT<sub>x</sub> MXene heterojunction has a reduced photo-generated carrier transfer resistance, showing improved photocatalytic HER performance of 10.11 mmol/g/h.



**Figure 12.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in photocatalyst. (a) Schematic illustration of fabrication procedure of CdS/Nb<sub>2</sub>CT<sub>x</sub>, (b) SEM image of CdS/Nb<sub>2</sub>CT<sub>x</sub>-40, (c) Photocatalytic H<sub>2</sub> evolution rates of CdS and CdS/Nb<sub>2</sub>CT<sub>x</sub>. Copyright Elsevier, 2022 [64].

The photocatalytic  $H_2$  evolution performance of  $Nb_2CT_x$  and its composites are listed in Table 4.

Materials	H <sub>2</sub> Production (µmol/g/h)	Sacrificial Reagent	References
Ag/Nb <sub>2</sub> O <sub>5</sub> @Nb <sub>2</sub> CT <sub>x</sub>	824.2	Methanol	[25]
1D CdS/2D Nb <sub>2</sub> CT <sub>x</sub>	5300	Lactic acid (10 vol%)	[26]
2D CdS/2D Nb <sub>2</sub> CT <sub>x</sub>	5040	Lactic acid (10 vol%)	[64]
$In_2S_3/Nb_2O_5/Nb_2C$	68.8	Methanol	[120]
Ru/Nb <sub>2</sub> O <sub>5</sub> @Nb <sub>2</sub> C	10,110	Methanol	[121]

Table 4. The specific capacities of  $Nb_2CT_x$  for Photocatalytic H<sub>2</sub> evolution.

#### 4.5. Sensors

MXene is also widely applied in sensing because of high conductivity, rich functional groups and high hydrophilicity.  $Nb_2CT_x$ , as another typical member of MXene family, has also generated wide interests for its potential use in sensors in recent years.

Zhao et al. [61] have designed an electrospinning neuron-like Nb<sub>2</sub>CT<sub>x</sub>/SA composite film, used as self-powered humidity sensors. The composite film can provide many nanochannels and adsorption sites for the directional ionization and transfer of water molecules. The moist-electric humidity sensor (MEHS) of Nb<sub>2</sub>CT<sub>x</sub>/SA composite shows fast response/recovery time, good long-term cycle stability, linearity, repeatability as well as low detection limit. Bi et al. [18] have studied the Nb<sub>2</sub>CT<sub>x</sub> in relative humidity (RH) measurement (Figure 13a). Microfibers and Nb<sub>2</sub>CT<sub>x</sub> are integrated using optical deposition for fiber-optic-based RH sensing. The RH sensing experiments show that as the RH levels increase from 18.5% to 72.4%, the transmission spectra (Figure 13b) initially blue shift with the sensitivity of -86 pm/% RH because of the change in effective refractive index of Nb<sub>2</sub>CT<sub>x</sub>. The transmission spectra (Figure 13c) exhibit a red shift in the RH from 72.4% to 95.4% RH, and the sensitivity is 585 pm/% RH due to the variations in Nb<sub>2</sub>CT<sub>x</sub> structure.



**Figure 13.** Applications of Nb<sub>2</sub>CT<sub>x</sub> in sensors. (a) Schematic diagram of the Nb<sub>2</sub>CT<sub>x</sub> nanosheet-coated microfiber RH sensor, (b) Transmission spectra in the RH range of 18.5-72.4% RH, (c) Transmission spectra in the RH range of 72.4-95.4% RH. Copyright American Institute of Physics, 2022 [18].

Li et al. [122] have synthesized a new heterogeneous convex fiber-tapered seven core fiber-convex fiber (CTC) probe via Nb<sub>2</sub>CT<sub>x</sub>, gold nanoparticles (AuNPs) and creatinase (CA) enzyme. The probe, combined with localized surface plasmon resonance (LSPR) technology, is used to test the concentration of creatinine. Since strong evanescent fields can induce the LSPR effect and stimulate AuNPs, the sensitivity of this probe increases. This sensor has a sensitivity of 3.1 pm/ $\mu$ M as well as limit of detection, which is 86.12  $\mu$ M at the linear from 0 to 2000  $\mu$ M. Wang et al. [28] have prepared a novel Nb<sub>2</sub>CT<sub>x</sub>/PANI sensor to efficiently detect NH<sub>3</sub> (Figure 14a). The gas-sensing response of the Nb<sub>2</sub>CT<sub>x</sub>/PANI-2 sensor (301.31%) is about 2.34 times higher than PANI sensor (128.81%), upto 100 ppm NH<sub>3</sub> under 87.1% RH (Figure 14b). The p-n junction is favorable to the enhancement of the gas-sensing properties of the Nb<sub>2</sub>CT<sub>x</sub>/PANI sensor.

In addition to the above applications,  $Nb_2CT_x$  sensors are also used for water quality monitoring [29],  $NO_2$  gas detection [123], fluorescence detection of heavy metal ions [27], biosensing application [56], and detection of human hemoglobin concentration [124], etc.



**Figure 14.** (a) Schematic illustration of fabrication process of the Nb<sub>2</sub>CT<sub>x</sub>/PANI sensor, (b) Response– concentration fitting curves of the PANI, Nb<sub>2</sub>CT<sub>x</sub>/PANI-1, Nb<sub>2</sub>CT<sub>x</sub>/PANI-2 and Nb<sub>2</sub>CT<sub>x</sub>/PANI-3 sensors toward 10–100 ppm NH<sub>3</sub>. Copyright Elsevier, 2020 [28].

# 4.6. Other Applications

4.6.1. Electromagnetic Interference (EMI) Shielding and Microwave Absorption (MA)

Nb<sub>2</sub>CT<sub>x</sub> is used in EMI shielding because of its unique structure, special metallic properties and abundant natural defects [33,125,126]. Rajavel et al. [126] have prepared a mixed composite of lamellar-structured Nb<sub>2</sub>CT<sub>x</sub> and one-step-extracted low-stratified Nb<sub>2</sub>CT<sub>x</sub> for EMI shielding. The composite material shows good EMI shielding efficiency of 44.09  $\pm$  1.99 dB at 12 GHz. Similarly, Nb<sub>2</sub>CT<sub>x</sub> has also been used for microwave absorption (MA) [127,128]. Song et al. [119] have synthesized Nb<sub>2</sub>O<sub>5</sub>/ Nb<sub>2</sub>CT<sub>x</sub> hybrids using microwave-assisted hydrothermal method. The hybrids shows good microwave absorption performance and a minimum reflection loss (RL) vof -44.1 dB at 2.8 GHz with the thickness of 5 mm.

# 4.6.2. Photodetector

Zhang et al. [67] have grown Bi NPs on the Nb<sub>2</sub>CT<sub>x</sub> surface to prepare Nb<sub>2</sub>CT<sub>x</sub>@Bi Schottky heterojunction. The Nb<sub>2</sub>CT<sub>x</sub>@Bi flexible photodetector has unique self-powered photodetection capability, and the response and recovery speed is both 0.08 s. In addition, Nb<sub>2</sub>CT<sub>x</sub>@Bi shows a specific detectivity of  $4.63 \times 10^{12}$  Jones and a responsivity of 585.25 mA/W. Because of the reduced charge transmission time as well as the Schottky barrier and built-in electric field at the interface of Bi and Nb<sub>2</sub>CT<sub>x</sub>, the uniflow spreading process of electrons is accelerated, the separation of photo-induced electron/hole pairs is facilitated and carrier recombination dynamics is hindered. Therefore, Nb<sub>2</sub>CT<sub>x</sub>@Bi shows improved photodetection capability.

# 4.6.3. Perovskite Solar Cells (PVSCs)

Due to adjustable optoelectrical properties and numerous surface functional groups,  $Nb_2CT_x$  has also been used in Perovskite solar cells [30,129]. Zhang et al. [129] have reduced the work function (WF) by replacing -F groups with -NH<sub>2</sub> groups via hydrazine treatment and obtained T-Nb<sub>2</sub>CT<sub>x</sub>. The T-Nb<sub>2</sub>CT<sub>x</sub> electron transport layer (ETL) shows outstanding optoelectrical properties, highly matched energy level with perovskite layer,

high transmittance and conductivity. After incorporating  $T-Nb_2CT_x$  into the perovskite precursor, hydrogen bond with iodine ions is formed to retard the crystallization rate. The  $T-Nb_2CT_x$  as ETL achieves a high power conversion efficiency (PCE) of 21.79% with good stability.

In addition to the above applications,  $Nb_2CT_x$  is also used in nitrogen reduction reaction (NRR) catalysts [130], dye adsorption [34], solar-driven water evaporation [131],  $H_2O_2$  synthesis catalysts and organic dyes degradation [132].

#### 5. Summary and Outlook

This review has summarized the synthesis and properties of Nb<sub>2</sub>CT<sub>x</sub> as well as its different applications in secondary batteries, supercapacitors, electrocatalysis and sensing. The wide application of Nb<sub>2</sub>CT<sub>x</sub> is attributed to its high conductivity, high theoretical specific capacity, abundant functional groups and high hydrophilicity. At present, the study of Nb<sub>2</sub>CT<sub>x</sub> is at the preliminary stage, as there are many challenges related to synthesis, properties and application which should be fixed before it can be studied further. This review also deals with some potential application such as EMI, MA and photodetector, that needs to be focused on.

In terms of synthesis, Nb<sub>2</sub>CT<sub>x</sub> is etched using mainly acid etching methods, especially the high concentration HF etching method, which is effective but harmful to the humans and the environment. Therefore, exploring fluorine-free etching methods is essential. Both molten salt etching and electrochemical etching have been reported as fluorine-free etching methods, but the experimental conditions and parameter details of these methods still need to be fully discussed. In addition, the delamination of Nb<sub>2</sub>CT<sub>x</sub> is a necessary step to get single-layer or few-layer Nb<sub>2</sub>CT<sub>x</sub> nanosheets. Most of the intercalation currently used are organic bases, such as TMAOH and TPAOH. Nonetheless, further exploration is needed to develop low-cost and harmless intercalants.

Because of the Van der Waals force between  $Nb_2CT_x$  sheets,  $Nb_2CT_x$  nanosheets tend to be aggregated or restacked to form a dense structure, sacrificing the specific surface area and active sites. When using as an electrode in energy storage, it will lead to poorer cycling stability, lower specific capacitance value and rate capability. Therefore, more work should be carried out to suppress the self-stacking of  $Nb_2CT_x$  in order to increase the specific surface area, provide more active sites, and optimize the electrochemical utilization of  $Nb_2CT_x$ .

Treatment of surface functional groups (alkali treatment or heat treatment) is an effective way to tune the property of Nb<sub>2</sub>CT<sub>x</sub>. Specially, increasing -O functional groups and reducing -F functional groups on Nb<sub>2</sub>CT<sub>x</sub> can greatly improve the electrochemical performance. Studies have shown that after treating with alkali (KOH, LiOH) or transition metal salts (CuSO<sub>4</sub>, FeSO<sub>4</sub>), the Nb<sub>2</sub>CT<sub>x</sub> or Nb<sub>2</sub>CT<sub>x</sub> composites tends to form a porous structure, with increasing specific surface area and more active sites. It is found that introducing carbon materials (CNTs and CQDs), conductive polymers, metal oxides or other metal cations into the Nb<sub>2</sub>CT<sub>x</sub>. In addition, single-atom doping, designing heterostructures, and constructing 3D porous structures are also effective methods to improve the electrochemical reaction active sites and enhance the electrochemical performance.

In terms of application, Nb<sub>2</sub>CT<sub>x</sub> is widely used in secondary batteries and supercapacitors. However, the mechanism of charge storage needs to be studied more in order to lay a solid foundation to guide future research or commercial application. Moreover, at present, there is no research on some areas of Nb<sub>2</sub>CT<sub>x</sub> applications. For example, aqueous zinc metal (Zn<sup>0</sup>) batteries (LAMBs) are among the most promising beyond-lithium technologies [133]. However, not many studies have been conducted to assess the application of Nb<sub>2</sub>CT<sub>x</sub> in LAMBs and as such it needs to be further carried out and developed. Additionally, application of Nb<sub>2</sub>CT<sub>x</sub> in the fields of biology, medicine, photoelectric detection and electromagnetic shielding also need more attention and effort. **Author Contributions:** Conceptualization, methodology, resources, data curation, writing, draft preparation, G.G. Supervision, project administration, funding acquisition and revision, F.G. All authors have read and agreed to the published version of the manuscript.

**Funding:** This work was funded by the National Natural Science Foundation of China, grant number 52002354, and China Postdoctoral Science Foundation, grant number 2020M672256.

**Data Availability Statement:** No new data were created or analyzed in this study. Data sharing is not applicable to this article.

Conflicts of Interest: The authors declare no conflict of interest.

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