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(Review Article)

Charge Storage in $Ti_3C_2T_x$ for Rechargeable Batteries: Recent Progress and Outlook

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Abstract

$Ti_3C_2T_x$ is a representative member of a rapidly growing family of two-dimensional, layered transition-metal carbides, nitrides, and carbonitrides whose electrochemical behavior is defined by three coupled features: (i) a highly conductive metal–carbon backbone, (ii) termination-rich surfaces that govern interfacial chemistry, and (iii) a stackable sheet morphology that creates an interlayer gallery where ions, solvent molecules, and intercalants can reside. Together, these attributes produce a material platform that is fundamentally different from many other 2D solids. Graphene, for example, offers exceptional conductivity and mechanical reinforcement but has a relatively inert basal plane unless defects or heteroatoms are introduced, whereas transition-metal dichalcogenides can provide layered insertion sites but may undergo phase evolution or conversion-type processes depending on voltage window. In contrast, $Ti_3C_2T_x$ combines metallic-like transport with intrinsically active, chemically tunable surfaces, allowing electrochemical function to be engineered through termination chemistry, interlayer spacing, and electrode architecture.

These characteristics have enabled $Ti_3C_2T_x$ to contribute to multiple battery platforms, including lithium-ion, sodium-ion, lithium sulfur, and zinc-based systems, where its role depends strongly on how it is assembled. In some cases, $Ti_3C_2T_x$ acts as the primary charge-storage host and delivers fast, surface-controlled storage with high power capability. In others, it serves as a conductive scaffold that lowers polarization in electrodes built from less conductive active phases, stabilizes microstructure during repeated volume changes, and improves electrolyte wetting through hydrophilic terminations. $Ti_3C_2T_x$ has also been used as a reinforcing component in hybrid electrodes, where it maintains electron percolation and reduces mechanical degradation, and as an interfacial modifier on current collectors, where it improves contact resistance and can regulate interphase formation. Across these functions, one of the most central and scientifically informative is its behavior as a charge-storage host material, because this role directly links atomic-scale surface chemistry and gallery structure to device-level capacity, rate capability, and stability.

In this review, I integrate recent progress in $Ti_3C_2T_x$ synthesis, processing, and characterization and evaluate, from a mechanistic perspective, how $Ti_3C_2T_x$ performs as an active electrode material in divalent-ion batteries. I focus on design approaches that improve reversible capacity, accelerate transport, and extend cycle life under multivalent charge carriers, where strong solvation and electrostatic interactions frequently impose kinetic and structural penalties. I compare the dominant storage pathways in single-phase $Ti_3C_2T_x$ with those in composite architectures and heterostructures, where graphene, carbon nanotubes, layered oxides, and other 2D materials are often introduced to prevent restacking, improve conductivity over practical thickness, and stabilize interfaces. Particular attention is given to how storage mechanisms evolve with electrolyte environment and operating window, including the balance between surface-controlled charging, pseudocapacitive redox at termination sites, and interlayer insertion or co-insertion phenomena that can involve solvent or protons in aqueous systems.

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Finally, I identify the main limitations that currently constrain practical deployment, including oxidation and moisture sensitivity, termination variability arising from synthesis history, restacking-driven transport losses, and the gap between laboratory-scale materials and scalable manufacturing. I outline research directions where microstructure engineering provides a realistic route to higher-performing divalent-ion storage, emphasizing controllable variables such as interlayer spacing and pillaring, flake size and alignment, defect populations, termination distributions, and engineered interfaces that reduce charge-transfer resistance while suppressing parasitic reactions. By framing $Ti_3C_2T_x$ alongside graphene and other established 2D materials, I highlight where $Ti_3C_2T_x$ offers unique advantages and where hybrid strategies are likely to be most effective for translating its intrinsic properties into durable, high-performance multivalent batteries.

Keywords: TI3C2TX; Divalent-Ion Batteries; Multivalent Charge Storage; Magnesium-Based Batteries; Zinc-Based Batteries; 2D Electrode Architectures; Microstructure Engineering

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1. Introduction

Large-scale deployment of solar and wind power has made the reliability problem of electricity more visible than ever: generation is variable, demand is time-dependent, and the grid requires tight control of frequency and voltage. These constraints make energy storage a system-level requirement rather than an optional add-on, because storage can time-shift renewable electricity, smooth short-term fluctuations, and support ancillary services such as peak shaving and fast response. In practice, this has driven rapid growth in electrochemical storage, which offers modularity, high energy density, and straightforward integration from portable electronics to grid-scale packs.1

Lithium-ion batteries remain the dominant technology because of their established manufacturing base and strong performance. However, concerns about supply-chain concentration, materials cost, and safety under high energy throughput have intensified the search for alternative battery chemistries. Two major directions have emerged.2 The first explores multivalent charge carriers, including Mg^{2+}, Zn^{2+}, Ca^{2+}, and Al^{3+}, motivated by the abundance of these elements and their high theoretical volumetric capacities. Despite this promise, multivalent systems often show limited practical performance because multiple barriers act simultaneously: slow solid-state transport, interfacial instability, limited compatibility with non-aqueous electrolytes, and progressive electrode damage that leads to capacity decay. A central kinetic limitation is the strong solvation and large effective size of multivalent ions in common solvents, which increases desolvation penalties and suppresses diffusion and charge-transfer rates at electrode interfaces. In addition, stronger Coulombic interactions can promote trapping at defects or surface terminations in host lattices, further reducing reversibility in many candidate electrodes.3

The second direction focuses on sodium and potassium batteries as lower-cost, resource-abundant complements to lithium. Sodium and potassium are among the most abundant elements in Earth's crust and each constitutes more than 2% of the lithosphere, supporting the case for scalable and economical supply. Their lower desolvation energies relative to multivalent ions can also benefit kinetics in suitable electrolytes. Yet Na^+ and K^+ storage remains limited by practical electrode issues, especially large volume changes during cycling that fracture particles, disrupt conductive networks, and accelerate electrolyte decomposition at freshly exposed surfaces. These degradation pathways reduce cycle life and can lower attainable energy density in full cells.4

Across both multivalent and monovalent alternatives, the bottleneck increasingly shifts to materials design: identifying electrode hosts and architectures that can simultaneously enable fast ion transport, stable interfaces, and mechanically tolerant microstructures over long cycling. This has motivated intensive research into new intercalation compounds, conversion and alloying anodes with engineered buffering strategies, porous and hierarchical electrodes that reduce diffusion length scales, and electrolyte formulations that form stable interphases. Together, these efforts aim to deliver cost-effective batteries with high reversible capacity, high-rate capability, and extended service life suitable for practical renewable-energy integration.5

A wide range of electrode materials and electrode-engineering strategies have been introduced to address recurring failure modes in rechargeable batteries, including thermal runaway, dendritic metal growth, and unstable interphase formation at the electrode–electrolyte interface. One commonly cited example is lithium titanate ($Li_4Ti_5O_{12}$, LTO), which is used as an anode to suppress lithium plating and reduce dendrite risk by operating at a higher potential than graphite. However, LTO's practical performance is limited by relatively low electronic conductivity and modest lithium-ion transport kinetics, which can constrain rate capability without additional conductive additives or microstructural optimization.6, 7

In parallel, high-capacity alloying anodes such as silicon have been widely studied because they can deliver low working potentials and substantially higher theoretical capacity than conventional intercalation anodes. Their primary limitation is the large volumetric expansion and contraction during cycling, which drives particle fracture, loss of electrical contact, and continuous interphase reformation. To accommodate this mechanical strain, nanostructured designs have been developed, including yolk–shell architectures in which silicon is spatially confined within a protective shell, often combined with conductive carbon frameworks such as graphene. These structures can improve capacity retention by buffering expansion and maintaining percolating electron pathways, but they may still suffer from reduced Coulombic efficiency due to persistent side reactions and irreversible lithium consumption associated with high surface area and interphase growth.7, 8

Cathode-side strategies have also expanded beyond single-phase materials. For instance, engineered nanocomposites have been explored to compensate for lithium inventory loss and improve high-rate behavior. Fe/LiF/Li_2O composite systems have been investigated as prelithiation-type cathode additives that can supply lithium and mitigate capacity fade associated with parasitic reactions, while also supporting power performance by improving charge-transfer pathways. Separately, polyanionic frameworks have attracted attention, particularly for sodium-ion batteries, because their strong covalent polyanion groups can stabilize the crystal lattice and provide a tunable redox environment. Olivine $NaFePO_4$ is one representative cathode with a theoretical capacity near 154 mA h g^{-1} and is often discussed as a sodium analogue of $LiFePO_4$. In practice, olivine $NaFePO_4$ is typically accessed through delithiation of $LiFePO_4$ followed by sodiation, which introduces processing complexity. Moreover, olivine $NaFePO_4$ is not the thermodynamically favored phase under many conditions, and its conversion toward the maricite polymorph is problematic because the maricite structure provides limited alkali-ion diffusion pathways, leading to low electrochemical activity.9

Over roughly the past twenty years, electrode research has expanded beyond inorganic intercalation compounds to include organic small molecules and redox-active polymers as deliberately designed charge-storage media. Interest in these materials is driven by the fact that their chemistry and microstructure can be tuned through synthesis, they can be produced from widely available feedstocks, and they offer pathways toward lower-toxicity and potentially recyclable electrodes.10 A representative example is microporous organic polymers (MOPs), which are attractive because their permanent porosity can generate very high internal surface areas, creating extensive electrolyte–electrode contact and increasing the number of accessible redox sites. This large interfacial area can improve utilization and rate response, although it can also increase the propensity for side reactions if the electrode–electrolyte chemistry is not well controlled.11

Organic radical batteries (ORBs) provide another approach, using stable radical moieties as redox centers to enable rapid electron-transfer reactions and, in many cases, strong cycling stability at high charge–discharge rates. A notable strategy is the post-synthetic crosslinking of poly (2,2,6,6-tetramethylpiperidinyloxy methacrylate) (PTMA) cathodes to improve mechanical integrity and maintain capacity at elevated rates in thick electrodes. Even when rate capability is strong, a recurring limitation of ORB-type materials is their relatively low intrinsic electronic conductivity, which stems from charge localization around radical groups; as a result, they often require conductive networks or architectural design to maintain efficient electron percolation through practical electrode thicknesses.12

In parallel with organic electrodes, two-dimensional nanomaterials have been widely examined as platforms for fast ion storage because they combine large accessible surface areas with short diffusion lengths and directionally open transport pathways.13, 14 From a structural standpoint, insertion hosts are often grouped by dimensionality: one-dimensional frameworks such as olivine-type channels, two-dimensional layered solids such as graphene-derived architectures, and three-dimensional networks such as silicon-based hosts. Layered, two-dimensional materials are frequently highlighted because ions can migrate between weakly bonded sheets with reduced transport distances compared with more tortuous three-dimensional pathways. An additional advantage is that their interlayer spacing can be adjusted through pre-synthetic design or post-synthetic intercalation of ions or molecules, which can expand gallery height, facilitate ion insertion, and improve tolerance to cycling-induced strain by reducing mechanical constraints during repeated (de)insertion.

Two-dimensional solids are also widely used as functional elements in composite electrodes, where they provide conductive pathways, mechanical reinforcement, and accessible ion-transport surfaces. For instance, Cui and co-workers reported a thermally processed architecture in which 3,4,9,10-perylenetetracarboxylic dianhydride (PTCDA) rods were embedded within a three-dimensional network formed by reduced graphene oxide (rGO) sheets, using roughly 9 wt% rGO. Relative to pristine PTCDA, the hybrid electrode delivered improved rate capability and cycling behavior, retaining 64% of its capacity at 1.0 A g^{-1} compared with its low-rate capacity (172 mA h g^{-1} at 0.1 A g^{-1}), corresponding to substantial gains in both delivered capacity and capacity retention versus the non-composite baseline.15, 16

Beyond their role in hybrids, pristine two-dimensional materials can function directly as active electrodes when their intrinsic conductivity and surface chemistry support rapid charge transport. Fang and co-workers, for example, developed a solution-deposition route to prepare mesoporous graphene nanosheets with high surface area, enabling extensive Li^+ adsorption at defect and edge sites and additional storage through intercalation pathways. More broadly, layered materials can also act as substrates that host other redox-active phases, improving electrical percolation and stabilizing microstructure during cycling. This “functional support” role is common in electrodes where the active component exhibits low conductivity or large strain, because a 2D scaffold can maintain electron pathways and reduce pulverization-driven contact loss.17

A diverse set of two-dimensional platforms has been explored for rechargeable batteries, including transition-metal dichalcogenides, phosphorene, hexagonal boron nitride, graphene and its derivatives, and $Ti_3C_2T_x$. $Ti_3C_2T_x$ is a layered transition-metal carbide that was first isolated in 2011 through selective removal of the Al-containing layers from Ti_3AlC_2 (a MAX phase), producing a two-dimensional Ti–C framework with surface terminations (T_x) introduced during synthesis. Since that initial report, $Ti_3C_2T_x$ has been studied intensively because it combines high electronic conductivity with large accessible surface area, solution processability in polar media, and tunable surface chemistry through controllable terminations and intercalation chemistry. These characteristics have supported applications spanning catalysis, photonics, electromagnetic interference shielding, membrane separations, and electrochemical energy storage.18

In energy-storage contexts, $Ti_3C_2T_x$ has attracted attention in both capacitive and battery-type devices. Its layered architecture provides short diffusion lengths and accessible galleries for ion insertion, while in situ measurements have shown that electrochemical cycling can proceed with comparatively small changes in interlayer spacing in certain electrolytes, which is advantageous for long-cycle operation when large crystallographic transformations are undesirable.19 For battery electrodes, theoretical calculations initially suggested favorable ion adsorption and insertion energetics, including Li^+ storage in $Ti_3C_2T_x$ accompanied by composition changes consistent with lithiated phases. Subsequent experimental studies expanded this concept to additional chemistries, and $Ti_3C_2T_x$ has been investigated in systems based on Na^+, K^+, Mg^{2+}, and Al^{3+} as well.20-22

In this review, I examine how $Ti_3C_2T_x$-based materials have been incorporated into rechargeable-battery electrodes across these chemistries. I first summarize synthesis routes and the key structural features that govern transport and interfacial behavior, then evaluate how $Ti_3C_2T_x$ functions as an active host, a conductive framework, and a composite component in full electrochemical cells, with emphasis on how composition, surface terminations, and microstructure influence charge-storage mechanism and device-level performance.23

2. Formation of $Ti_3C_2T_x$ from Layered Precursors: Chemistry and Structure

$Ti_3C_2T_x$ belongs to a broader family of two-dimensional transition-metal carbides, nitrides, and carbonitrides that are commonly described by the composition $M_{n+1}X_nT_x$. In this notation, M is an early transition metal, X is carbon and or nitrogen, n typically ranges from 1 to 3, and T_x denotes surface terminations introduced during synthesis. In practice, T_x most often represents a mixture of functional groups such as O-, OH-, and F- that decorate the outer metal surfaces and strongly influence electronic structure, wettability, and interfacial chemistry. This surface chemistry is a major reason these materials behave differently from other layered solids whose basal planes are largely chemically inert.24

The growth of interest in two-dimensional materials accelerated after graphene highlighted how reducing a solid to atomic or few-atom thickness can produce unusual combinations of transport and surface properties. That momentum quickly broadened to include many other 2D systems, including graphene derivatives, transition-metal dichalcogenides (for example MoS_2 and WS_2), phosphorene, layered oxides, and hexagonal boron nitride. Each class offers a distinct balance of conductivity, redox activity, and chemical stability. Graphene is often the conductivity and mechanical reinforcement benchmark in composites, while many TMDs provide layered insertion sites but can be limited by phase changes or conversion-type reactions under deep cycling. In this landscape, $Ti_3C_2T_x$ has become a heavily studied platform because it can combine high electronic conductivity with tunable surface chemistry and strong interaction with polar solvents.25

These layered carbides are typically produced by selectively removing the A-layer from a three-dimensional precursor known as a MAX phase. MAX phases follow the general formula $M_{n+1}AX_n$, where M and X carry the same meaning as above and A is commonly a group 13 or 14 element. Selective etching targets the A-layer while preserving the M-X slabs, transforming a bulk laminated crystal into a stack of 2D sheets that can be delaminated into single or few-layer flakes. Fluoride-containing chemistries are commonly used for this step, including HF and other fluoride-based etchants, and the choice of etchant, reaction conditions, and post-treatment strongly affects both the extent of delamination and the resulting termination chemistry. Intercalants used during delamination further tune layer spacing, flake dispersibility, and defect density, which together control properties relevant to electrochemical and interfacial applications.26-31

Structurally, many features of the parent MAX lattice are retained after etching. The resulting 2D sheets preserve a hexagonal arrangement within the basal plane and, when viewed from above, exhibit a sixfold symmetry reminiscent of graphene's lattice, although the chemistry is fundamentally different because the surfaces are terminated by T_x groups. In most fluoride-based routes, terminations form as a direct consequence of the etching and exfoliation environment rather than through a separate functionalization step, which distinguishes $Ti_3C_2T_x$ from many other 2D materials where surface chemistry is introduced later through controlled reactions. Because of this, the precursor composition, etching protocol, and intercalation history collectively determine the final stoichiometry, termination distribution, and defect landscape, and those variables can shift conductivity, hydrophilicity, and ion-transport behavior.18

The MAX-phase family itself is large and continues to grow, with many experimentally verified compositions, including widely studied examples such as Ti_2AlC and Ti_3AlC_2. These parent materials are often described as "hybrid" solids because they can exhibit ceramic-like attributes (such as high-temperature stability) alongside metallic characteristics (such as electrical and thermal conductivity). 32 That combination helps explain why their derived 2D counterparts are attractive for applications that require both chemical resilience and efficient charge transport, and it also clarifies why graphene is frequently paired with $Ti_3C_2T_x$ in composites: graphene contributes an ultraconductive, mechanically resilient scaffold, while $Ti_3C_2T_x$ contributes chemically active, termination-rich surfaces that couple strongly to ions, solvents, and polymer binders.33

3. $Ti_3C_2T_x$: Synthesis Routes and Structural Characteristics

Single-layer $Ti_3C_2T_x$ flakes are most commonly produced by a top-down route that can be viewed as three coupled stages: (i) preparing a layered precursor, (ii) selectively removing the A-layer, and (iii) delaminating the etched solid into few-layer or single-layer sheets.34 This workflow has remained the dominant preparation strategy since the first demonstrations in 2011 because it reliably converts a bulk laminated crystal into dispersible 2D building blocks.

The process starts with synthesis of a three-dimensional, layered precursor, most often a MAX phase (though related non-MAX layered carbides and nitrides have also been explored). In these parent crystals, strong M–X slabs are separated by comparatively reactive A-element layers, creating a natural "weak plane" that can be chemically removed. The resulting 2D framework can be described as alternating (n+1) metal layers and n carbon and or nitrogen layers, and this geometry is important because it exposes the outer metal surfaces during etching. Those newly exposed surfaces readily bind functional groups from the etching environment, which is why $Ti_3C_2T_x$ and related materials typically emerge with mixed terminations rather than a single well-defined surface chemistry.35

Selective etching is then used to extract the A-layer while largely preserving the M–X backbone, leveraging the contrast between strong M–X bonding and weaker, more reactive M–A interactions. After removal of the A-element, the product is a multilayer stack in which individual $Ti_3C_2T_x$ sheets remain aligned but are separated by expanded galleries and held together primarily by weak interlayer interactions. At this stage, the material often resembles an "accordion-like" morphology and contains intercalated species and terminations that influence spacing, hydrophilicity, and electronic transport.36, 37

Delamination converts these multilayer stacks into colloidally stable suspensions of few-layer and, under optimized conditions, predominantly single-layer flakes. This step commonly relies on intercalants and mechanical energy (for example agitation or sonication) to increase gallery spacing and overcome interlayer adhesion while maintaining lateral flake integrity. The same concept is widely used across 2D materials. Graphene oxide is separated by oxidation and exfoliation followed by reduction to restore conductivity, while layered transition-metal dichalcogenides are often exfoliated chemically (for example with lithium intercalation) or mechanically to access thinner sheets. A key difference for $Ti_3C_2T_x$ is that its termination chemistry is introduced during the etch itself, so delamination conditions can further shift the termination distribution, defect density, and interlayer chemistry, all of which later affect electrochemical performance.38

In the earliest demonstration of this approach, Ti_3AlC_2 was etched to remove Al, and the reaction pathway was presented as a stepwise removal of the A-layer accompanied by formation of surface terminations on the remaining Ti–C framework.

In MAX-phase precursors, the bonding within the M–X slabs is dominated by strong directional interactions that are commonly described as mixed covalent and ionic in character, leading to high cohesion within each carbide and or nitride layer. By comparison, the M–A interactions are more metallic in nature and substantially weaker. This contrast in bond strength is the fundamental reason selective etching works: the chemistry can disrupt the A-containing layers while leaving the M–X framework largely intact.39

During A-layer removal, cleavage occurs preferentially at the weaker M–A interfaces. As the A atoms are extracted, the exposed transition-metal surfaces become undercoordinated and highly reactive. They are therefore rapidly passivated by species present in the etching environment, generating the surface terminations collectively labeled T_x. In fluoride-containing routes, these terminations typically include O-, OH-, and F-, and their relative abundance depends on the etchant composition, water content, temperature, and reaction time. This termination step is not a separate functionalization stage; it is intrinsically coupled to the etching process and directly shapes electronic conductivity, wettability, and interfacial charge transfer.10, 40

After etching, the product is usually a multilayer stack rather than isolated monolayers. Converting these stacks into single-to-few-layer flakes requires delamination, which can be achieved chemically (through intercalation that expands the interlayer galleries) and or mechanically (through agitation, shearing, or sonication that overcomes residual adhesion).7 This exfoliation logic parallels other 2D systems. For example, graphene oxide is delaminated by introducing oxygenated groups that increase interlayer spacing and electrostatic repulsion, whereas many transition-metal dichalcogenides are thinned by ion intercalation or liquid-phase exfoliation. In $Ti_3C_2T_x$, delamination efficiency is particularly sensitive to intercalant choice and to how terminations and trapped species modify interlayer interactions.22, 41, 42

Post-etch washing is a practical but critical step. Residual acids, dissolved salts, and reaction byproducts must be removed to bring the suspension toward a moderately acidic to near-neutral range, often around pH 5 to 6, both for safe handling and for improving colloidal stability. Washing also helps limit continued parasitic reactions that can alter surface terminations or accelerate oxidation during storage. Importantly, the etching reaction rarely consumes all of the starting precursor. Unreacted MAX grains and partially etched intermediates are common, meaning the overall yield can be substantially below quantitative conversion and the final product may contain a mixture of fully converted flakes and residual precursor unless additional separation steps are used.18, 43

Soon after the first reports in 2011, most synthetic routes produced $Ti_3C_2T_x$ and related compositions primarily as stacked, multilayer solids obtained by direct HF-based etching. These early products confirmed that selective removal of the A-layer could convert a laminated 3D precursor into a termination-bearing layered carbide, but the materials were still dominated by restacked sheets rather than freely dispersed 2D flakes.44 The situation changed around 2013, when intercalation-based delamination was shown to separate the stacks into single- and few-layer nanosheets. In that approach, large organic species were inserted between adjacent layers, increasing gallery spacing and weakening intersheet attraction so that the material could be exfoliated into atomically thin flakes. This step effectively opened the door to a broad toolbox of intercalants and delamination aids, enabling reproducible preparation of colloidal suspensions and allowing researchers to probe thickness-dependent properties and build more controlled electrode architectures.

It is also important that exfoliation does not always yield only flat nanosheets. Under strong mechanical processing, electron microscopy has revealed curled morphologies such as scrolls and tube-like structures, indicating that bending and edge stresses, together with termination chemistry and interlayer interactions, can drive shape transformations. Similar behavior is seen in other 2D systems: graphene and graphene oxide can form scrolls under sonication, and certain transition-metal dichalcogenides can roll or fold depending on defect distributions and strain gradients. These morphological pathways matter because they change accessible surface area, ion transport geometry, and electrical percolation in films and composites.45

As synthesis moved from proof-of-concept to materials engineering, attention shifted to defects and surface terminations because both are strongly coupled to performance. Atomic-scale disorder, including vacancies and adatoms, has been observed after aggressive etching and high-energy ultrasonication. While some defects can originate in the parent precursor, a substantial fraction is introduced during the chemical removal of the A-layer and subsequent delamination. In practical terms, this means that synthesis conditions can inadvertently trade yield for structural quality, and this tradeoff resembles what is commonly encountered in other 2D materials. For example, chemical oxidation routes for graphene oxide promote exfoliation but also introduce high defect densities that reduce conductivity, while harsh intercalation protocols used to exfoliate layered dichalcogenides can generate phase changes and edge disorder. For $Ti_3C_2T_x$, controlling these defects is particularly important because they influence flake conductivity, oxidation susceptibility, mechanical integrity, and electrochemical stability.46

These challenges motivated gentler separation strategies designed to reduce mechanical damage. A key development was the minimally intensive layer delamination (MILD) concept, which generates HF in situ from a lithium fluoride salt, enabling controlled etching and intercalation-assisted delamination without the need for strong sonication. This approach made it easier to access larger, higher-quality flakes and improved scalability by simplifying processing and increasing reproducibility. Related in situ HF strategies have also used other fluoride salts, with the underlying goal of controlling reaction rate, termination chemistry, and delamination efficiency in a more predictable manner.47

Defects are only part of the synthesis problem, however. Termination chemistry is equally critical, particularly because F-containing terminations are common in fluoride-based routes and can be unfavorable for certain applications. In electrochemical systems, relatively inert fluorine terminations can reduce the density of active sites, alter wettability, and increase interfacial resistance, while also raising environmental and safety concerns associated with HF-centered processing. These issues have accelerated efforts toward fluorine-reduced and fluorine-free routes. One important direction is electrochemical etching, where selective removal of the A-layer is driven by applied potential in aqueous electrolytes, providing an alternative pathway that can offer high yield while reducing reliance on HF-like chemistry. More broadly, the trajectory mirrors the evolution seen in graphene manufacturing: as the field matured, emphasis shifted from any method that produces flakes to methods that deliver controlled chemistry, low defect density, and scalable reproducibility.42

4. Properties of $Ti_3C_2T_x$ and Related Layered Carbides

MXene are known for the unique and excellent mechanical, electrical, optical and thermal $Ti_3C_2T_x$ and related 2D transition-metal carbides show a combination of mechanical, electrical, optical, and thermal characteristics that is uncommon in a single material class. A central reason is that their properties are not set only by the underlying Ti–C lattice, but also by surface terminations (T_x), defect density, interlayer species (water and ions), and flake stacking or alignment in the final architecture. This sensitivity is one way they differ from graphene, whose basal plane is largely chemically uniform, and from many transition-metal dichalcogenides (TMDs) such as MoS_2, where the dominant variability is thickness, phase, and defect concentration rather than dense, synthesis-imposed terminations.

Mechanical response is particularly termination-dependent. Computational studies have consistently predicted that O-rich terminations increase stiffness, while OH- and F-rich surfaces reduce elastic constants relative to O-terminated surfaces. Experiments support the broader idea that functionalization changes both modulus and failure behavior: functionalized sheets can show reduced Young's modulus but higher strain tolerance than non-functionalized counterparts. In other words, terminations can soften the lattice while simultaneously improving deformability, which is relevant for flexible electrodes and freestanding films. Composition also matters across the broader $Ti_{n+1}C_n$ family. First-principles comparisons across Ti-based carbides often identify Ti_2C as among the stiffest members, with reported elastic constants that can exceed those of widely studied layered materials such as MoS_2. In practical electrode constructs, however, the measured modulus also reflects microstructure, including flake size distribution, porosity, interflake junction density, and polymer or electrolyte content, so intrinsic stiffness does not automatically translate to mechanically strong films unless assembly is controlled.

Chemical stability and oxidation behavior are equally important because they control shelf life, processing windows, and electrochemical performance. $Ti_3C_2T_x$ is susceptible to oxidation in the presence of oxygen and water, and degradation can proceed under ambient conditions or in pressurized aqueous environments. Reported outcomes include formation of TiO_2 nanocrystals (anatase and or rutile), sometimes accompanied by carbon-rich residue, yielding TiO_2–C hybrid structures. The spatial distribution of TiO_2 can vary with conditions, ranging from embedded nanocrystals within carbonaceous matrices to more uniform decoration across $Ti_3C_2T_x$ sheets. This variability is not just a materials curiosity. Oxidation modifies conductivity, changes surface chemistry, alters interlayer spacing, and can either degrade electrochemical performance or, in some composite contexts, introduce new functionalities. Similar stability challenges exist across other 2D materials. Phosphorene readily oxidizes in air, many TMDs can undergo edge oxidation or phase evolution, and graphene oxide is chemically reactive unless reduced and stabilized. For $Ti_3C_2T_x$, the practical implication is that termination chemistry, storage conditions, and processing atmospheres must be treated as design parameters rather than procedural details.

From an electronic-structure standpoint, un-terminated $Ti_{n+1}X_n$ frameworks are often predicted to be metallic, but the degree of metallicity can decrease as n increases due to changes in bonding and band structure. Nitride analogues are typically more metallic than carbide analogues because of their electron count and bonding characteristics. Once terminations are introduced, the picture becomes more tunable: surface groups can shift band alignment, open or narrow band gaps, and modify density of states near the Fermi level, producing behavior that spans metallic to semiconducting depending on termination identity and arrangement.48, 49 This kind of "chemistry-controlled electronics" contrasts with graphene, where band structure is strongly tied to lattice symmetry and doping, and it complements TMDs, where semiconducting band gaps are common but tunability is frequently achieved through thickness, strain, and phase control rather than termination chemistry. Strain engineering can also be relevant for certain terminated compositions, where tensile strain modifies interatomic distances and can reduce band gap, indicating that mechanical deformation and electronic response can be coupled.50

Electrically, $Ti_3C_2T_x$ films can exhibit high conductivity when flakes are well delaminated, oxidation is suppressed, and interflake contacts are optimized. High conductivity is often attributed to the metallic character of the Ti–C backbone and the presence of transition-metal conduction pathways, but measured values are strongly architecture-dependent. Factors such as flake alignment, restacking density, junction resistance, residual intercalants, and termination distribution can shift conductivity by orders of magnitude. This is one reason graphene is frequently incorporated into $Ti_3C_2T_x$ composites: graphene can reduce junction resistance and improve percolation, while $Ti_3C_2T_x$ contributes ion-accessible galleries and chemically active surfaces. Conversely, pairing $Ti_3C_2T_x$ with semiconducting TMDs can balance conductivity and redox activity, while h-BN can be used as a chemically stable dielectric barrier in layered heterostructures when electrical insulation and mechanical reinforcement are desired.

Overall, the defining feature of $Ti_3C_2T_x$ is that its "properties" are not a single fixed set of numbers. They emerge from a coupled structure–chemistry–assembly relationship, where terminations and microstructure are as important as the underlying 2D lattice.51

5. Electrochemical Behavior of $Ti_3C_2T_x$ Electrodes

$Ti_3C_2T_x$ is electrochemically distinctive because its charge storage is governed by a coupled set of structural features: a layered gallery that can host ions and solvent, electrically conductive basal planes that support fast electron transport, and termination-rich surfaces that define interfacial chemistry.52 The stacked-sheet architecture introduces interlayer spacing as a first-order design variable. When the galleries are sufficiently open and wettable, ions can enter and leave with relatively short diffusion lengths, enabling high-rate operation in both battery-type and capacitive regimes. In contrast, when sheets restack tightly or become partially blocked by trapped species, transport becomes more tortuous and rate capability often decreases.52

Intercalation is therefore not merely a synthesis step but a tool for engineering electrochemical function. Expanding the interlayer galleries with bulky intercalants can increase electrolyte accessibility and reduce kinetic limitations, and specific agents can produce substantially larger spacings than as-prepared stacks. 53Larger gallery heights are frequently associated with higher gravimetric and volumetric capacitance because a greater fraction of the internal surface area becomes electrochemically addressable and ion transport barriers are reduced. At the same time, excessive swelling or poorly controlled intercalation can weaken mechanical cohesion in films, increase electronic junction resistance, and accelerate oxidation, so the best performance usually comes from controlled expansion rather than maximal expansion.54

A key advantage of $Ti_3C_2T_x$ relative to many other 2D materials is that its surface terminations provide a direct handle on interfacial charge storage. Termination identity and density influence wettability, ion desolvation, charge-transfer resistance, and the potential window in a given electrolyte. This is fundamentally different from graphene, where the basal plane is chemically inert and charge storage relies heavily on double-layer capacitance unless defects or heteroatom doping are introduced.55 It also complements transition-metal dichalcogenides, where electrochemical activity may involve deeper structural changes, phase evolution, or conversion processes depending on voltage. For $Ti_3C_2T_x$, even modest shifts in termination chemistry can alter whether storage is dominated by surface-controlled processes, interlayer insertion, or mixed mechanisms, and these shifts often depend on electrolyte composition and water content.

Because electrolyte interactions are central, $Ti_3C_2T_x$ performance cannot be discussed independently of the electrolyte environment. Surface redox activity and ion adsorption can modify local electrolyte structure, influence interphase formation, and in some cases catalyze side reactions that reduce Coulombic efficiency or narrow the stable operating window. This is especially important when comparing aqueous and non-aqueous systems, or monovalent and multivalent ions, where solvation strength and desolvation barriers differ substantially. In practical devices, optimizing electrolyte formulation and controlling water and impurity levels are therefore as important as controlling the flake structure, because the electrolyte determines which terminations are stable and which charge-storage pathways are accessible.

To improve electrochemical outcomes, most successful approaches combine chemistry control with architectural design. Surface functionalization is used to tune wettability and electronic structure, hybridization introduces complementary phases that improve conductivity or stabilize structure, and composite formation prevents restacking while preserving ion-accessible porosity. Graphene and carbon nanotubes are common conductive partners that reduce junction resistance and maintain percolation in thick electrodes, while integrating $Ti_3C_2T_x$ with layered oxides or TMDs can introduce additional redox capacity and broaden the usable voltage range. Across these strategies, the underlying design goal is consistent: decouple ion transport from electronic transport by keeping galleries accessible for ions while maintaining continuous conductive pathways for electrons, and simultaneously stabilize the surface chemistry that controls interfacial reactions.

6. Other Applications

Aqueous zinc-based energy storage is receiving sustained attention because it aligns with practical constraints that often limit deployment of large battery systems: low materials cost, nonflammable electrolytes, and comparatively safe handling. Zinc metal is particularly attractive because it can deliver high theoretical capacity and operates at a relatively negative redox potential in water-based electrolytes, enabling competitive cell voltages without relying on highly reactive alkali metals. 56 These advantages have encouraged rapid growth in zinc-ion batteries and zinc-ion capacitors, and they also explain why 2D materials are increasingly investigated as both active hosts and functional interfaces in Zn systems.57

Recent work has expanded how $Ti_3C_2T_x$ is viewed in Zn electrolytes. For years, Zn^{2+} storage in $Ti_3C_2T_x$ was often interpreted as being largely surface-controlled, dominated by adsorption and desorption rather than deep insertion. In many aqueous zinc salts, the electrochemical signature of conventionally etched $Ti_3C_2T_x$ is close to an ideal capacitive response, with a near-rectangular CV and weak or absent redox peaks. This behavior is consistent with double-layer charge storage plus a modest pseudocapacitive contribution at termination sites. By contrast, in less protic or organic environments, faradaic features can become more apparent, indicating that the electrolyte strongly governs which sites are active and whether insertion-like processes are kinetically accessible. A further complication in aqueous Zn systems is that Zn^{2+} storage can couple to proton chemistry. Studies using delaminated carbide electrodes, including architectures stabilized with carbon nanotubes, have provided evidence for co-participation of Zn^{2+} and H^+ during cycling, meaning that the measured capacity can reflect mixed carriers unless the mechanism is carefully resolved.58

From a device-engineering perspective, Zn systems face persistent stability challenges that are distinct from many lithium-based cells. Compared with alkaline Zn–MnO_2 chemistries, rechargeable Zn-ion configurations in aqueous electrolytes can still suffer from zinc hydroxide and oxide formation, parasitic hydrogen evolution, and non-uniform metal deposition that generates dendrites. These processes shorten cycle life and raise failure risks. One historical mitigation strategy has been shifting toward neutral or mildly acidic electrolytes (for example $ZnSO_4$-type solutions), which can reduce dendritic deposition and suppress some irreversible byproduct formation, although anode corrosion and interfacial side reactions remain major bottlenecks.

Cathode performance introduces an additional constraint: Zn^{2+} is strongly solvated and divalent, so its interaction with many host lattices is electrostatically intense, which can slow diffusion and destabilize frameworks during insertion. To compensate, cathode designs frequently rely on conductive scaffolds and composite architectures. Carbon materials, especially graphene and reduced graphene oxide, are commonly paired with MnO_2 to provide percolating electron pathways and reduce polarization. In the same spirit, other 2D materials such as MoS_2 and related TMDs have also been examined as conductive or pseudocapacitive partners, although they can introduce their own structural evolution depending on voltage range.

$Ti_3C_2T_x$ adds a different set of composite advantages relative to conventional carbons. Its high electrical conductivity, strong hydrophilicity, dense packing (beneficial for volumetric metrics), and termination-rich surfaces can improve electrolyte wetting and interfacial charge transfer. These features make $Ti_3C_2T_x$ particularly effective as a functional framework for MnO_2 and other oxide cathodes, where it can simultaneously address low conductivity and mechanical degradation. Three-dimensional $Ti_3C_2T_x$ architectures, including porous and flower-like assemblies, are especially useful because they preserve open electrolyte pathways while maintaining continuous conductive networks. When coupled with highly electroactive MnO_2, such structures can improve both structural stability and electronic transport during repeated cycling, which translates to better rate performance and longer cycle life.

Overall, in aqueous Zn-based devices, $Ti_3C_2T_x$ is most impactful when it is treated as an interface and architecture material rather than only a standalone active phase. Its strongest benefits often appear in composites where graphene or CNT networks enhance long-range conductivity while $Ti_3C_2T_x$ provides ion-accessible, termination-engineered surfaces and a mechanically resilient scaffold that stabilizes oxide cathodes under Zn^{2+} insertion stress

7. Recommendations and Research Priorities

$Ti_3C_2T_x$ and related 2D carbides have proven to be versatile electrochemical materials, with demonstrated relevance in Zn-, Mg-, and Ca-based storage systems and in broader electrochemical technologies. Even with rapid progress, several practical barriers still limit translation from strong lab-scale demonstrations to reliable, scalable devices. In my view, the highest-impact next steps fall into three linked areas: synthesis modernization, mechanism-guided performance design, and stability engineering.58-60

First, synthesis remains a bottleneck because many widely used preparation routes rely on HF-centered chemistries. These protocols are effective but introduce safety risks, handling complexity, and waste-management burdens that are difficult to justify at scale. Future work should continue shifting toward fluoride-free or fluorine-reduced approaches that maintain high yield while improving environmental compatibility and process control. Electrochemical etching and other non-HF strategies are promising because they can decouple etching from harsh chemical conditions and potentially provide tighter control over termination chemistry and defect formation. Scalable production will also require precursor-to-flake quality control analogous to what enabled graphene manufacturing to mature: standardized metrics for oxidation state, termination distributions, flake size, and impurities, paired with process windows that reproducibly deliver target properties.61

Second, the electrochemical signature of $Ti_3C_2T_x$ electrodes often reflects predominantly surface-controlled storage, which supports fast kinetics and high power but can limit energy density when voltage profiles are sloped and faradaic utilization is modest. This is not inherently a weakness. It makes $Ti_3C_2T_x$ highly competitive for high-rate architectures such as hybrid ion capacitors and for roles as conductive, ion-accessible frameworks. However, when the goal is higher energy density, the design strategy should shift from “using $Ti_3C_2T_x$ alone” to “using $Ti_3C_2T_x$ as a platform.” Compositional tuning of terminations and controlled interlayer chemistry can activate additional storage sites, but the more general route is hybridization with higher-capacity phases. Here, lessons from other 2D materials are useful. Graphene is rarely the main capacity contributor in batteries, but it is a powerful conductivity and mechanical scaffold. Similarly, $Ti_3C_2T_x$ can be paired with redox-active oxides, sulfides, Prussian blue analogues, or organic carbonyl compounds to raise capacity while maintaining rapid electron transport and stable electrode integrity. Layered TMDs can also serve as complementary redox partners in some voltage windows, provided that phase evolution and conversion pathways are controlled.62

Third, stability is a decisive issue. $Ti_3C_2T_x$ is sensitive to oxygen and water, and oxidation can restructure surfaces, reduce conductivity, and destabilize interlayer architecture. Improving durability requires more than simply storing materials carefully. It requires deliberate stabilization strategies that preserve electrochemical accessibility while suppressing degradation. Practical routes include surface passivation that does not block ion transport, thin protective coatings, antioxidant and oxygen-scavenging additives in inks and slurries, and encapsulation methods for films and electrodes. The field can borrow from stabilization approaches used for other reactive 2D materials. Phosphorene, for example, pushed development of encapsulation and barrier-layer strategies, while TMD research emphasized controlling edge chemistry and defect reactivity. For $Ti_3C_2T_x$, stabilization should be evaluated not only by shelf-life but also by how it affects interfacial resistance, termination stability, and cycling-induced structural changes.63

Overall, the path to practical impact is clear. Replace hazardous synthesis with scalable, controllable routes; use $Ti_3C_2T_x$ as an enabling conductive and ion-accessible platform rather than forcing it to deliver all capacity alone; and implement stabilization strategies that protect the material without sacrificing ion transport. If these priorities are addressed, $Ti_3C_2T_x$-based electrodes and composites can become strong candidates for next-generation aqueous and multivalent systems, while also expanding into device formats where graphene and other 2D materials have already proven the value of architecture-driven electrochemical design.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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