

MXenes: Two-Dimensional Building Blocks for Future Materials and Devices



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Since the synthesis of Ti_3C_2 was reported in 2011,¹ we have seen tremendous growth in research on synthesis, characterization, and applications of two-dimensional (2D) carbides and nitrides named MXenes. It was, in fact, an article in *ACS Nano* in 2012 that reported the syntheses of M_2X , M_3X_2 , and M_4X_3 and announced the birth of an entirely new large family of 2D materials.² M_5C_4 was reported in *ACS Nano* in 2020, further increasing the structural diversity of 2D carbides and carbonitrides. The general formula of MXenes is $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M represents a transition metal, X represents carbon or nitrogen, n can be from 1 to 4, and T_x indicates terminations on the surface of the outmost transition metal layers (Figure 1).³ The possibility of in-plane and out-of-plane ordering of the metal atoms brings the number of possible structures to well over 100.⁴ Surface terminations increase it by another order of magnitude, while the possibility of forming solid solutions on M and X sites, as well as mixed terminations, leads to a potentially unlimited number of 2D materials with distinct properties.

ACS Nano is one of the leaders in publishing cutting-edge research on MXenes and guiding the research community through Editorials and Perspectives,^{5–7} which outline future research directions. The number of papers on MXenes that we publish increases every year, proportional to the number of submissions received. For example, 18 papers reporting research on MXenes appeared in *ACS Nano* in the first 3 months of 2021. This virtual issue highlights many of the key findings in the field that have been published in *ACS Nano*. Of course, due to length limitations, we could only include a small portion of all the excellent MXene papers from our journal in this virtual issue. We emphasize the most cited and most frequently accessed recent publications, representing various methods of MXene synthesis, processing, property characterization, and applications. In this Editorial accompanying the virtual issue, we introduce the variety of MXene compositions; describe their unique place in the materials world, their fundamental properties, and the parameters governing them; briefly summarize syntheses and control of surface terminations of MXenes; and discuss their promise in selected applications, as well as challenges in the field.

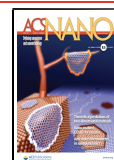
■ PLACE OF MXENES IN THE MATERIALS WORLD

The discovery of fascinating properties in single- and few-layer graphene attracted attention to many other 2D materials.^{8,9} After initial studies focusing on exfoliation of other van der

Waals bonded solids, such as BN or transition metal dichalcogenides,¹⁰ 2D silicon and germanium structures that do not have van der Waals bonded layered precursors were demonstrated.^{11–13} MXenes—2D carbides and nitrides of transition metals that are produced by selective etching of strongly bonded layered solids, such as MAX phases—followed in 2011.¹ Why do MXenes stand out among so many already available 2D materials? MXenes provide 2D sheets with metallic electrical conductivity, a property that was largely missing from the palette of 2D materials, most of which are semiconductors, semimetals, or dielectrics. Moreover, MXenes offer reported conductivity values up to 20,000 S/cm,¹⁴ higher strength and stiffness compared to other solution-processed 2D materials, scalable solution syntheses (kg batches), sufficient environmental stability for a large variety of applications, biocompatibility, and aqueous solution processing without surfactants. One can consider them to be hydrophilic, water-dispersible 2D metals or electrically conductive clay (MXenes' rheological properties are similar to that of clay).¹⁵ There are dozens of MXenes already available, and a potentially infinite number of compositions is possible. Moreover, further control of properties can be achieved by reversible chemical/electrochemical intercalation, applied potential, or illumination. MXenes show metallic conductivity, but their Fermi levels can be tuned by external stimuli, almost like in semiconducting materials.

Those combinations of properties of MXenes are valuable for many applications. A combination of conductivity and redox ability enables energy storage, conductivity and catalytic ability allow electrocatalysis, conductivity plus transparency are needed for transparent conductors and heaters, conductivity combined with color enables photonic and optoelectronic devices, tunable plasmon resonance can be used in photo-thermal therapy, photocatalysis, and surface-enhanced Raman spectroscopy.¹⁶

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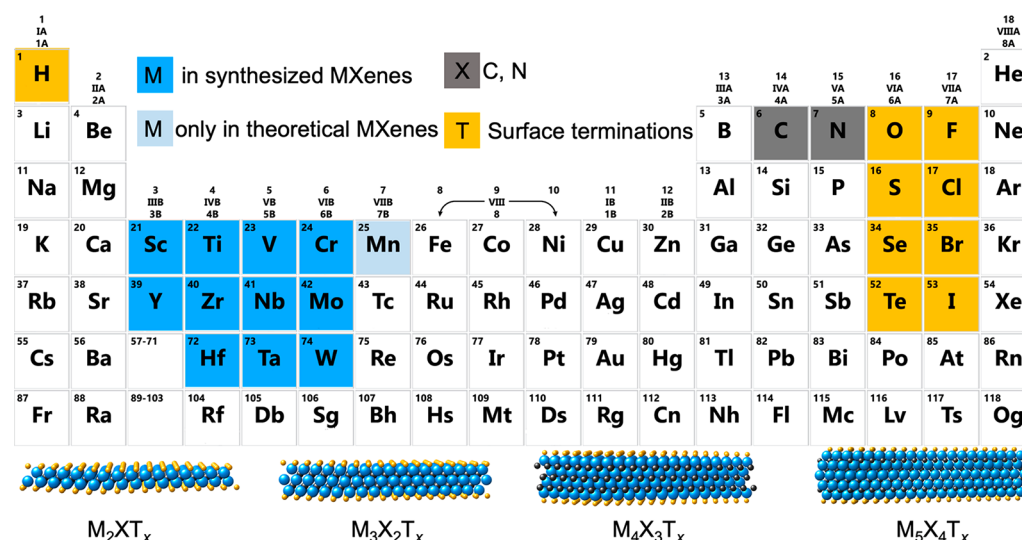


Figure 1. Periodic table showing compositions of MXenes. Elements used to build MXenes are color-coded. The schematics of four typical structures of MXenes are presented at the bottom. Courtesy of Prof. Babak Anasori, UIPUI.

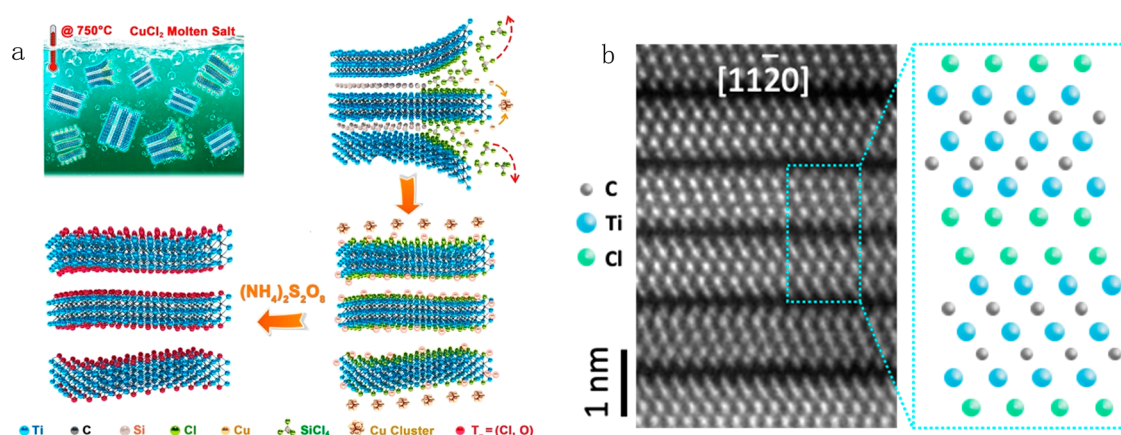


Figure 2. (a) Schematic of $Ti_3C_2T_x$ MXene preparation by molten salt etching. Ti_3SiC_2 MAX phase is immersed in $CuCl_2$ Lewis molten salt at $750^\circ C$. The reaction between Ti_3SiC_2 and $CuCl_2$ results in the formation of $Ti_3C_2T_x$ MXene. Molten-salt stacked $Ti_3C_2T_x$ MXene is obtained after further washing in ammonium persulfate solution. Reprinted with permission from ref 24. Copyright 2020 Springer Nature. (b) High-resolution scanning transmission electron microscopy image showing the atomic positions of $Ti_3C_2Cl_2$. Cl atoms terminate the surface of $Ti_3C_2Cl_2$. Reprinted from ref 22. Copyright 2019 American Chemical Society.

■ EXPANSION OF THE MXENE FAMILY VIA SURFACE TERMINATIONS

Physical and chemical properties of MXenes are strongly influenced by the composition and configuration of surface terminations, as shown in numerous theoretical and experimental studies. When the A-site atoms of the MAX phase are etched, the freshly exposed and unsaturated transition metal atoms are immediately coordinated by anions in the etchant, forming the surface terminations T_x of $M_{n+1}C_nT_x$ MXenes. Therefore, the surface chemistry of MXene has been a mainstream research topic, and it is expected to receive even more attention in the future.

The first MXene, $Ti_3C_2T_x$, was produced using hydrofluoric acid (HF) etching of Ti_3AlC_2 in 2011, and its surface terminations were a mixture of $-O$, $-OH$, and $-F$.^{1,17} The relative stability of these surface terminations follows the order of $Ti_3C_2O_2 > Ti_3C_2F_2 > Ti_3C_2(OH)_2$.^{18,19} *Ab initio* calculations of electronic properties of MXenes indicate that all bare MXene sheets are metals with a high density of states at the Fermi level and a high concentration of carriers. However,

oxygen and some other surface moieties can open the band gap and transform them into semiconductors, depending on the kind and position of terminations.²⁰ Relations between surface chemistry and the work function, conductivity, transition to a superconducting state, magnetism, and energy storage capacity of various MXenes have been established computationally and experimentally. For example, Li-ion storage capacity is strongly dependent on the nature of the surface terminations, with oxygen termination exhibiting the highest theoretical capacity.²¹

However, producing MXenes with uniform terminations remained a challenge until the Huang group reported on etching MAX phases in $ZnCl_2$ molten salts, which produced MXenes with exclusively chlorine terminations ($Ti_3C_2Cl_2$ and Ti_2CCl_2 ; Figure 2).^{22,23} This approach was further developed into a general Lewis-acidic-melt etching method that is applicable to various systems.²⁴ The wide range of constituents of Lewis-acidic melts implies the feasibility of tuning surface terminations of MXenes and expanding their chemistry. A series of halogenated $Ti_3C_2T_x$ ($T = Br, I$, or their combination)

MXenes were further synthesized through the Lewis-acidic-melt etching route.²⁵ These new terminations result in excellent electrochemical properties, particularly in zinc-ion battery cathodes.²⁶ Talapin *et al.* performed etching of MAX phases in CdBr_2 melt to obtain bromide-terminated MXenes and further substituted bromine with oxygen, sulfur, selenium, tellurium, and NH groups, as well as produced defunctionalized MXene surfaces.²⁷ The surface terminations affect all physical properties of MXenes; for example, surface-group-dependent superconductivity was exhibited by Nb_2C MXenes. The molten salt technique enables synthesis of fully oxygen terminated MXenes and, therefore, may lead to experimental verification of the predicted semiconducting MXenes, 2D ferromagnets, and topological insulators, which may find applications in highly efficient thermoelectrics, magnetic storage devices, and quantum computing technology. These examples show how property- and application-oriented design of MXenes can result in the advancement of the field, in terms of both understanding their fundamental properties and also paving the way for their use in next-generation technologies.

There are currently more than 30 different experimentally made stoichiometric MXenes and more than 100 (not considering surface terminations) theoretically predicted compositions with distinct electronic, physical, and (electro)-chemical properties. In addition, solid solutions on M and X sides are possible, and the possibility of having multiple single (O, Cl, F, S, *etc.*) or mixed (O/OH/F) surface terminations makes MXenes a large and diverse family of 2D materials.²⁸

■ THE RISE OF MXENES AND FUTURE CHALLENGES

Initially, the interest in MXenes was primarily for energy storage applications, and these included Li^+ , Na^+ , K^+ , Mg^{2+} , Al^{3+} , Li-S, Na-S, and K-S batteries, supercapacitors, and hybrid energy storage devices such as Li-ion and Na-ion capacitors.²⁹ MXenes are promising as charge hosts due to their 2D galleries for fast transport of ions; metallic electronic conductivity; reversible redox reactions of the surface transition metal layers; and spontaneous intercalation of cations, including multivalent ions. Easy solution processing is convenient for processing electrode films or creating complex electrode architectures. MXenes have been used in printable electrodes of on-chip and textile/wearable energy storage devices and also for passive components, such as current collectors preventing dendrites growth, binders for Si, and carbon anodes. The next step was capacitive deionization, which uses supercapacitor principles for removing salt ions from water. Electrocatalysis for hydrogen and oxygen evolution reactions (HER and OER), CO_2 reduction, and ammonia synthesis followed. Implantable brain and epidermal electrodes as well electrochromic devices equally benefit from the high conductivity and tunable optoelectronic properties of MXenes.^{30,31} Although exploration of those and other biomedical applications of MXenes only began about 5 years ago, this is a quickly expanding field because MXenes may enable life-saving medical treatments and replacement of expensive noble metals.

During the initial period of exploration, the MXene family kept growing and new MXenes showed a variety of properties, opening new applications and resulting in a surge of research activities that led to fast expansion of the field. 2017 was the beginning of MXenes' "gold rush". The first International Conference dedicated entirely to MXenes in 2018 confirmed that a new field of materials research had been established.

Since then, the world of 2D carbides and nitrides has been growing at an unprecedented rate. Currently, MXenes are experiencing an expansion of their applications diversity and an explosion in the number of publications and patents. There were three international meetings and symposia solely dedicated to MXenes in the second half of 2020, with the MXene 2020 conference at Drexel University bringing together more than 2000 registered participants. However, due to the COVID-19 pandemic, only the third International Conference on MXenes on October 11–14 was able to be held in a hybrid, and not fully online, format. It was organized by the Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, and attracted ~500 attendees from China, the United States, the United Kingdom, France, Germany, Switzerland, Sweden, and many other countries. Compared to the first and second International Conferences on MXenes, held at Jilin University in 2018 and Beijing University of Chemical Technology in 2019, respectively, the topics presented in 2020 broadened and deepened the MXene research interests and application fields. Conference sessions included "MXene/MAX/MAB Synthesis & Characterization & 4th Symposium on Synthesis and Application of Novel Material", "MXene for Energy Storage", "MXene for Optical, Electrical and EMI Devices", "MXene for Catalysis, Environment and Sensors", and "MXene Theoretical Simulation". The sessions focused on the state of art and key issues for MXene research and development, as well as the future opportunities and challenges in the field. A group of leading experts in the field, including conference chairs Q. Huang and Y. Gogotsi, as well as P. Simon, B. Anasori, M. Naguib, M. Barsoum, P. Eklund, P. Persson, and J. Rosen with their students and postdocs, assembled a list of open questions in the field, and all conference participants were offered an opportunity to prioritize them. Table 1 presents this list with the priority decreasing from questions 1 to 32.

The availability of numerous two-dimensional materials led to a paradigm shift from exploring single "wonder materials" to assembling materials and devices from hundreds or thousands of low-dimensional building blocks with properties required for advancing technology.

By addressing the issues listed in Table 1, the nanoscale research community can make new discoveries and enable novel applications of MXenes in many fields. Emerging related structures, such as 2D borides (MBenes), vapor-grown 2D nitrides, mechanically exfoliated MAX phases (MAXenes), and predicted MC_2 2D carbides,³² will further expand the field of 2D non-oxide materials and provide numerous building blocks for future technologies. The realization of theoretically predicted exciting properties of MXenes,³³ ranging from tunable superconductors to 2D ferromagnets, topological insulators, and energy-efficient electrocatalysts may lead to great technological advances.

Table 1. Research Challenges for the Next Decade, as Ranked by MXene Researchers

- 1 develop environmentally friendly, safe, efficient, and scalable synthesis methods
- 2 utilize 2D MXenes as nanoscale building blocks to develop three-dimensional nanoarchitectures, including vertically aligned, hybrid, and other structures
- 3 improve chemical and temperature stability of MXenes
- 4 electronic, optical, magnetic, thermal, and thermoelectric properties and quantum confinement effect
- 5 control surface chemistry
- 6 develop large-scale, large-area single-crystal MXene films
- 7 develop self-assembly techniques to prepare MXene films with aligned flakes and controlled orientation/distance between flakes
- 8 explore MXenes beyond $\text{Ti}_3\text{C}_2\text{T}_x$
- 9 develop various MXenes with terminations other than O, OH, or halogens (such as S, Se, Te, H, N, etc.) and methods to switch between them
- 10 understand the effects of confined ions and molecules (mobility and bonding) between the MXene sheets on MXenes properties and performance in different applications
- 11 understand charge storage mechanisms of MXenes in electrochemical energy storage in aqueous and nonaqueous electrolytes
- 12 transfer the outstanding mechanical properties of MXenes to ceramic, metal, and polymer matrix composites
- 13 engineer defects to control properties
- 14 develop termination-controlled physical properties (such as superconductivity)
- 15 understand the health and environmental safety/toxicity of MXenes with different transition metals and surface terminations
- 16 explore the syntheses of other materials using MXenes as one of the precursors
- 17 develop delamination protocols for MXenes beyond $\text{Ti}_3\text{C}_2\text{T}_x$ without the use of organic intercalants that will provide improved conductivity and other properties
- 18 synthesize MXenes from non-Al MAX phases
- 19 synthesize MXenes with no surface functional groups in vacuum by chemical vapor deposition or physical vapor deposition
- 20 understand interflake charge transport mechanism in MXene films and determine how to control it
- 21 understand the mechanisms of electromagnetic wave attenuation in MXenes
- 22 understand and reach the theoretical limits of properties of MXenes
- 23 expand the chemical space of X in MXene from C to N and B
- 24 develop theoretical methods for reliable predictions and understanding of novel MXene
- 25 develop novel two-dimensional transition metal carbides, nitrides, and borides: TiC_2 , MoN, Ti_2B , TiB_2 , etc.
- 26 demonstrate band gaps in MXenes and synthesize semiconducting MXenes
- 27 demonstrate magnetic MXenes
- 28 identify new precursors (beyond MAX phases)
- 29 determine the roles of the precursor's structure/defects/stoichiometry on the characteristics of MXenes
- 30 develop high-entropy MXenes and the arrangement of constitutive elements
- 31 synthesize nontitanium based MXenes without the direct use of hydrofluoric acid (MILD)
- 32 standardize MXene characterization procedures

■ FROM MXENE TO TWO-DIMENSIONAL HYBRIDS AND BEYOND

Ultrathin 2D materials exhibit versatile advantageous properties. Their electronic properties range from metallic to semimetal, semiconducting, and insulating. Their atomic thickness offers maximum mechanical flexibility and optical transparency. Large lateral size and atomic thickness enable high surface areas available for surface reactions. The availability of numerous 2D materials led to a paradigm shift

from exploring single “wonder materials” to assembling materials and devices from hundreds or thousands of 2D building blocks with properties required for advancing technology.^{34,35}

Assembly of hybrid materials enables combinations of properties that no conventional (single) material can provide. Great diversity of properties can be achieved using various 2D flakes. Tunable properties of responsive materials enable sensing and actuation, color change, and controlled interactions with electromagnetic waves over a wide range of frequencies. Extremely anisotropic materials become possible when 2D flakes are aligned. One can build hybrid and composite materials using self-assembly or additive manufacturing techniques. Device assembly can be done without waste. Manufacturing of complex shapes and integration with zero-dimensional and one-dimensional materials into three-dimensional materials and structures becomes possible. Catalytically and redox-active particles and organic molecules, 2D oxides, MoS_2 , graphene, phosphorene, nanotubes, conducting polymers, and other nanomaterials have been integrated with MXenes.^{36–38} However, there remain numerous challenges to be overcome, including large-scale programmable and controllable self-assembly, matching work functions to control junctions between dissimilar materials, controlling interaction/bonding between the components, and selecting materials that offer the exact combinations of properties required for specific applications. Help from machine learning and artificial intelligence will be required to make this vision a commercial reality.



Yury Gogotsi, Associate Editor  orcid.org/0000-0001-9423-4032



Qing Huang  orcid.org/0000-0001-7083-9416

■ AUTHOR INFORMATION

Complete contact information is available at:

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Notes

Views expressed in this editorial are those of the authors and not necessarily the views of the ACS.

Corresponding authors: Y.G. gogotsi@drexel.edu, Q.H. huangqing@nimte.ac.cn.

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