

# The Chemistry Of Transition Metal Carbides And Nitrides

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2D Metal Carbides and Nitrides (MXenes)  
The Thermodynamic Properties of Metal Carbides and Nitrides  
Transition Metal Carbides and Nitrides  
Synthesis and transport properties of 2D transition metal carbides (MXenes)  
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MXenes  
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Surface characterization of 2D transition metal carbides (MXenes)  
Capacitive Performance of Two-Dimensional Metal Carbides  
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refractory materials volume 7 transition metal carbides and nitrides discusses the developments in transition metal carbide and nitride research this volume is organized into nine chapters that emphasize the mechanical and superconducting properties of these compounds the introductory chapters deal with the general properties preparation techniques characterization crystal chemistry phase relationships and thermodynamics of transition metal carbides and nitrides the following chapter highlights the mechanical properties of these compounds such as elastic and plastic deformation fracture strengthening mechanisms and hardness the discussion then shifts to specific electrical and magnetic properties including electrical resistivity hall coefficient and magnetic susceptibility a separate chapter is devoted to carbides and nitrides as superconductors the concluding chapters explore certain theories that explain the mechanisms of band structure and bonding in carbides and nitrides this volume is of great value to research workers in metallurgy ceramics physics chemistry and related fields as well as to advanced students investigating problems concerning high temperature materials or interstitial compounds

this book arose from a symposium titled transition metal carbides and nitrides preparation properties and reactivity organized by jae sung lee masatoshi nagai and myself the symposium was part of the 1995 congress of pacific rim chemical societies held in honolulu hawaii between december 17 22 1995 the meeting was the first major conference to exclusively address the theme of metal carbides and nitrides and brought together many of the major researchers in the field over 50 scientists and engineers reported their latest findings in five sessions of presentations and discussions the book closely follows the topics covered in the conference theory of bonding structure and composition catalytic properties physical properties new methods of preparation spectroscopy and microscopy the book is unique in its coverage it provides a general introduction to the properties and nature of the materials but also covers their latest applications in a wide variety of fields it should thus be of interest to both experts and nonexperts in the fields of material science solid state chemistry physics ceramics engineering and catalysis the first chapter gives an overview and many of the chapters

provide summaries of advanced topics all contributions were peer reviewed

this book describes the rapidly expanding field of two dimensional 2d transition metal carbides and nitrides mxenes it covers fundamental knowledge on synthesis structure and properties of these new materials and a description of their processing scale up and emerging applications the ways in which the quickly expanding family of mxenes can outperform other novel nanomaterials in a variety of applications spanning from energy storage and conversion to electronics from water science to transportation and in defense and medical applications are discussed in detail

since the isolation and characterization of graphene there has been a growing interest in 2d materials owing to their unique properties compared to their 3d counterparts recently a family of 2d materials of early transition metal carbides and nitrides labelled mxenes has been discovered  $\text{Ti}_2\text{CTx}$   $\text{Ti}_3\text{C}_2\text{Tx}$   $\text{Mo}_2\text{TiC}_2\text{Tx}$   $\text{Ti}_3\text{CNTx}$   $\text{Ta}_4\text{C}_3\text{Tx}$   $\text{Ti}_4\text{N}_3\text{Tx}$  among many others where t stands for surface terminating groups o oh and f mxenes are mostly produced by selectively etching a layers where a stands for group a elements mostly groups 13 and 14 from the max phases the latter are a family of layered ternary carbides and or nitrides and have a general formula of  $\text{Mn}_n\text{X}_{n-1}\text{X}_3$  where m is a transition metal and x is carbon and or nitrogen the produced mxenes have a conductive carbide core and a non conductive o oh and or f terminated surface which allows them to work as electrodes for energy storage applications such as li ion batteries and supercapacitors prior to this work mxenes were produced in the form of flakes of lateral dimension of about 1 to 2 microns such dimensions and form are not suitable for electronic characterization and applications i have synthesized various mxenes  $\text{Ti}_3\text{C}_2\text{Tx}$   $\text{Ti}_2\text{CTx}$  and  $\text{Nb}_2\text{CTx}$  as epitaxial thin films a more suitable form for electronic and photonic applications these films were produced by hf  $\text{NH}_4\text{HF}_2$  or lif hcl etching of magnetron sputtered epitaxial  $\text{Ti}_3\text{AlC}_2$   $\text{Ti}_2\text{AlC}$  and  $\text{Nb}_2\text{AlC}$  thin films for transport properties of the ti based mxenes  $\text{Ti}_2\text{CTx}$  and  $\text{Ti}_3\text{C}_2\text{Tx}$  changing n from 1 to 2 resulted in an increase in conductivity but had no effect on the transport mechanism i e both  $\text{Ti}_3\text{C}_2\text{Tx}$  and  $\text{Ti}_2\text{CTx}$  were metallic in order to examine whether the electronic properties of mxenes differ when going from a few layers to a single flake similar to graphene the electrical characterization of a single  $\text{Ti}_3\text{C}_2\text{Tx}$  flake with a lateral size of about 10  $\mu\text{m}$  was performed these measurements the first for mxene demonstrated its metallic nature along with determining the nature of the charge carriers and their mobility this indicates that  $\text{Ti}_3\text{C}_2\text{Tx}$  is inherently of 2d nature independent of the number of stacked layers unlike graphene where the electronic properties change based on the number of stacked layers changing the transition metal from ti to nb viz comparing  $\text{Ti}_2\text{CTx}$  and  $\text{Nb}_2\text{CTx}$  thin films the electronic properties and electronic

conduction mechanism differ  $\text{Ti}_2\text{CTZ}$  showed metallic like behavior resistivity increases with increasing temperature unlike  $\text{Nb}_2\text{CTZ}$  where the conduction occurs via variable range hopping mechanism vrh where resistivity decreases with increasing temperature furthermore these studies show the synthesis of pure  $\text{Mo}_2\text{CTZ}$  in the form of single flakes and freestanding films made by filtering  $\text{Mo}_2\text{CTZ}$  colloidal suspensions electronic characterization of free standing films made from delaminated  $\text{Mo}_2\text{CTZ}$  flakes was investigated showing that a vrh mechanism prevails at low temperatures 7 to 60 K upon vacuum annealing the room temperature rt conductivity of  $\text{Mo}_2\text{CTZ}$  increased by two orders of magnitude the conduction mechanism was concluded to be vrh most likely dominated by hopping within each flake other Mo based mxenes  $\text{Mo}_2\text{Ti}_2\text{CTZ}$  and  $\text{Mo}_2\text{Ti}_2\text{C}_3\text{TZ}$  showed vrh mechanism at low temperature however at higher temperatures up to rt the transport mechanism was not clearly understood therefore a part of this thesis was dedicated to further investigating the transport properties of Mo based mxenes this includes  $\text{Mo}_2\text{CTZ}$  out of plane ordered  $\text{Mo}_2\text{Ti}_2\text{CTZ}$  and  $\text{Mo}_2\text{Ti}_2\text{C}_3\text{TZ}$  and vacancy ordered  $\text{Mo}_1\text{33CTZ}$  magneto transport of free standing thin films of the Mo based mxenes were studied showing that all Mo based mxenes have two transport regimes a vrh mechanism at lower temperatures and a thermally activated process at higher temperatures all Mo based mxenes except  $\text{Mo}_1\text{33CTZ}$  show that the electrical transport is dominated by inter flake transfer as for  $\text{Mo}_1\text{33CTZ}$  the primary electrical transport mechanism is more likely to be intra flake the synthesis of vacancy ordered mxenes  $\text{Mo}_1\text{33CTZ}$  and  $\text{W}_1\text{33CTZ}$  raised the question of possible introduction of vacancies in all mxenes vacancy ordered mxenes are produced by selective etching of Al and Sc or Y atoms from the parent 3d max phases such as  $\text{Mo}_2\text{3Sc132AlC}$  with in plane chemical ordering of Mo and Sc however not all quaternary parent max phases form the in plane chemical ordering of the two M metals thus the synthesis of the vacancy ordered mxenes is restricted to a very limited number of max phases i present a new method to obtain mxene flakes with disordered vacancies that may be generalized to all quaternary max phases as proof of concept i chose NbC mxene as this 2d material has shown promise in several applications including energy storage photothermal cell ablation and photocatalysts for hydrogen evolution starting from synthesizing  $\text{Nb}_2\text{3Sc132AlC}$  quaternary solid solution and etching both the Sc and Al atoms resulted in  $\text{Nb}_1\text{33C}$  material with a large number of vacancies and vacancy clusters this method may be applicable to other quaternary or higher max phases wherein one of the transition metals is more reactive than the other and it could be of vital importance in applications such as catalysis and energy storage

the present disclosure relates to a method for producing a metal carbide where the method includes thermally treating a

molecular precursor in an oxygen free environment such that the treating produces the metal carbide and the molecular precursor includes  $M_nX_x$  where m is the metal of the metal carbide n includes nitrogen or a nitrogen containing functional group and x is between zero and six inclusively

since their discovery in 2011 mxenes 2d carbides nitrides and carbonitrides of early transition metals have developed into one of the largest and most intensively studied families of 2d materials they offer unique properties and are being explored in a large variety of applications this book compiles the most important research from a pioneer of the field professor yury gogotsi and his interdisciplinary research team as well as numerous collaborators worldwide it reports on the discovery and rise of mxenes and describes their synthesis and processing properties and incorporation into polymer ceramic and metal matrices to produce composites it also discusses the potential of mxenes for use in energy storage optics electronics and sensing as well as biomedical environmental and electrocatalysis applications the book will appeal to anyone interested in nanomaterials and their synthesis properties and applications

a comprehensive overview of the synthesis of high quality mxenes in transition metal carbides and nitrides mxenes handbook synthesis processing properties and applications a team of esteemed researchers provides an expert review encompassing the fundamentals of precursor selection mxene synthesis characterizations properties processing and applications you ll find detailed discussions of the selection of mxene members for specific applications as along with summaries of the physical and chemical properties of mxenes including electrical mechanical optical electromechanical electrochemical and electromagnetic properties the authors delve into both successful and unsuccessful synthesis examples offering detailed explanations of various failures to facilitates a comprehensive understanding of the reasons behind unsuccessful syntheses additionally they provide detailed examinations on the characterizations of mxenes empowering readers to develop a sophisticated understanding of how to achieve optimal quality flake size oxidation states and more you ll also find a thorough review of common applications of mxenes including electrochemical applications electromagnetic interference shielding communications devices and more comprehensive explorations of solution and non solution processing of mxenes practical discussions of the synthesis of high quality mxene powders colloidal solutions and flakes including information about mxene precursors fulsome treatments of mxene precursor selection and their impact on mxene quality tailored to meet the needs of graduate students researchers and scientists in the areas of materials science inorganic chemistry and physical chemistry the transition metal carbides and nitrides mxenes handbook will also benefit

biochemists and professionals working in drug delivery

refractory carbides and nitrides are useful materials with numerous industrial applications and a promising future in addition to being materials of great interest to the scientific community although most of their applications are recent the refractory carbides and nitrides have been known for over one hundred years the industrial importance of the refractory carbides and nitrides is growing rapidly not only in the traditional and well established applications based on the strength and refractory nature of these materials such as cutting tools and abrasives but also in new and promising fields such as electronics and optoelectronics

research on two dimensional 2d materials is a rapidly growing field owing to the wide range of new interesting properties found in 2d structures that are vastly different from their three dimensional 3d analogues in addition 2d materials embodies a significant surface area that facilitates a high degree of surface reactions per unit volume or mass that is imperative in many applications such as catalysis energy storage energy conversion filtration and single molecule sensing mxenes constitute a family of 2d materials consisting of transition metal carbides and or nitrides which are typically formed after selective etching of their 3d parent max phases the latter are a family of nanolaminated compounds that typically follow the formula  $M_nA_x$  where m is a transition metal a is a group 13 or 14 element and x is c and or n selective etching by aqueous f containing acids removes the a layer leaving 2d  $M_n$  slabs instantly terminated by a mix of o oh and f groups the first and most investigated mxene is  $Ti_3C_2Tx$  where tx stands for surface termination which has shown record properties in a range of applications eg electrode in li batteries supercapacitors sieving membrane electromagnetic interference shielding and carbon capture adding to that over 30 different mxenes have been discovered since 2011 exhibiting alternative or superior properties most importantly elegant routes for property design in the mxene family has been demonstrated by means of either varying the chemistry in the  $M_nA_x$  compound by alloying two m elements or by changing the structure of the mxene by introducing vacancies the present work has a led to an additional route for post synthesis property tuning in mxenes by manipulation of surface termination elements this enables a unique toolbox for property tuning which is not available to other 2d materials and is highly beneficial for applications that is dependent on surface reactions furthermore chemical and structural characterization of terminations on single sheets is essential to rule out the influence of intercalants or contamination that is typically present in multilayer mxene samples or thin films for that purpose a method for preparing isolated contamination free single sheets of mxene

samples for transmission electron microscopy (TEM) characterization was established in order to determine vacancy and termination sites atomically resolved scanning TEM imaging and image simulations was carried out. Two main processes were employed to substitute the termination elements: 1) an initial thermal treatment in vacuum facilitates F desorption and it was shown that O terminations rearrange on the evacuated sites. H<sub>2</sub> gas exposure in a controlled environment demonstrated a removal of the remaining O terminations as a result. Termination-free MXene is possible to realize under vacuum conditions. 2) CO<sub>2</sub> was introduced as a first non-inherent termination on MXene by in situ CO<sub>2</sub> gas exposure at low temperatures that was a first demonstration of Ti<sub>3</sub>C<sub>2</sub>TX as promising material for carbon capture. Additionally, O-saturated surfaces were demonstrated after introduction of O<sub>2</sub> gas on the F-depleted Ti<sub>3</sub>C<sub>2</sub>TX MXene, which is highly relevant for hydrogen evolution reactions where fully O-terminated Ti<sub>3</sub>C<sub>2</sub>TX are predicted to improve efficiency. A Lewis acid melt synthesis method was used to realize the first MXene exclusively terminated with Cl. Moreover, this was the first report of a MXene directly synthesised with terminations other than O, OH, and F. Furthermore, we have expanded the space of property tuning by introduction of chemical ordering by selective etching of Y in an alloyed Mo<sub>2</sub>Y<sub>1-3</sub>C<sub>2</sub>TX MXene. This either produced chemical ordering with one M (Mo) element and vacancies or ordering between two M (Mo) and Y elements. This was further reported to significantly increase volumetric capacitance because of the increased number of active sites around vacancies leading to an increasing charge density. As a final note, the stability of Nb<sub>2</sub>C<sub>2</sub>TX MXene under ambient conditions was investigated. It was found that the surface Nb adatoms present after etching got oxidized over time, which resulted in local clustering and effectively degraded the MXene. This work has demonstrated reproducible surface characterization methods for determining termination elements and sites in 2D MXenes that is ultimately governing MXene properties. Most importantly, we report on a new approach for MXene property tuning as well as contributing to several existing property tuning approaches.

Recently, a new family of two-dimensional 2D early transition metal carbides and carbonitrides called MXenes was discovered. Unlike graphene, whose chemistry is restricted to carbon, MXenes allow a variety of chemical compositions and are establishing themselves as a large new class of two-dimensional materials. MXenes combine the metallic conductivity of transition metal carbide layers with the hydrophilic nature of their mostly hydroxyl or oxygen-terminated surfaces. In essence, they behave as conductive clays and have shown much of promise as electrode materials for Li-ion batteries. Prior to the initiation of this study, there have been no reports on the capacitive properties of MXenes. In this work, the

potential was explored of the new family of the two dimensional carbides mxenes as electrode materials for electrochemical capacitors this study was focused on  $\text{Ti}_3\text{C}_2\text{Tx}$  it was established that variety of single and multiply charged cations such as  $\text{Li}$ ,  $\text{Na}$ ,  $\text{K}$ ,  $\text{NH}_4^+$ ,  $\text{Mg}^{2+}$  can intercalate mxenes chemically or electrochemically and participate in energy storage highly reversible electrochemical insertion of the same cations has been demonstrated for  $\text{Ti}_3\text{C}_2\text{Tx}$  in aqueous electrolytes perfect capacitive behavior was observed for  $\text{Ti}_3\text{C}_2\text{Tx}$  mxene even at quite high charge and discharge rates all coupled with excellent cyclability no drop in capacitance was observed even after 10 000 cycles further investigation showed that surface chemistry has significant effect on the resulting capacitance i.e. by creating predominantly oxygen containing functionalities the capacitance can be substantially boosted in comparison to the as received material it was also demonstrated that  $\text{Ti}_3\text{C}_2\text{Tx}$  clay produced using  $\text{LiF}$   $\text{HCl}$  mixture with predominantly oxygen containing functionalities showed outstanding capacitance up to 900  $\text{F cm}^{-3}$  and can be manufactured in to electrodes in less than 10 min without need of binder or conductive additive electrochemical in situ xas measurements detected changes in  $\text{Ti}$  oxidation state during cycling which matched closely the observed experimental values of the material's capacitance therefore it was concluded that mechanism of electrochemical storage of the  $\text{Ti}_3\text{C}_2\text{Tx}$  mxene clay is predominantly pseudocapacitive also concept of all solid state asymmetrical supercapacitor freestanding and current collector free based on  $\text{Ti}_3\text{C}_2\text{Tx}$  was developed among other applications using in situ afm the potential of the use of mxenes in electrochemical actuators was demonstrated it was also shown that mxenes other than  $\text{Ti}_3\text{C}_2\text{Tx}$  also demonstrated a lot of promise for electrochemical capacitors  $\text{Nb}_2\text{C}_2\text{Tx}$   $\text{CNT}$  paper electrodes showed high volumetric capacitance of 325  $\text{F cm}^{-3}$  when tested in a  $\text{Li}$  ion capacitor configuration

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