

Process Safety Analysis for $\text{Ti}_3\text{C}_2\text{T}_x$ MXene Synthesis and Processing

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Supporting Information

ABSTRACT: MXenes are two-dimensional metal carbides with promising applications in energy storage and sensors. Guidelines for safe, scalable MXene synthesis are important due to ongoing efforts to scale-up production of these novel nanomaterials. Hazard identification before scale-up will allow for inherently safer designs prior to actual implementation. Here, we investigate hazards associated with MXene production, including MAX phase synthesis from raw materials, etching of the MAX phase to MXene clay, exfoliation to MXene nanosheets, and postprocessing of MXenes with $\text{Ti}_3\text{C}_2\text{T}_x$ as a model species. The major hazards in MXene synthesis are the potential for dust ignition, runaway reactions, and toxic chemical exposure. Because the synthesis of MXenes is a multistep process, safety guidelines for each step are evaluated, including preventive and mitigating measures, best practices, and emergency procedures and responses. This includes handling of combustible powders, exothermic reactions, and harsh chemical etchants. This study is intended to facilitate safer MXene synthesis across various levels of scale-up, from large laboratory batches to commercial production.



1. INTRODUCTION

Nanomaterials are unique because they often present safety hazards in both synthesis and health. Over the last several decades, a number of studies on biological and health hazards of nanomaterials have been published, and these studies are crucial in understanding the effects of nanomaterials on humans and the environment.^{1,2} Despite these recent advances, there is a stark absence of research assessing the process safety of nanomaterials synthesis and scale-up. The number of novel nanomaterials synthesized by researchers and small-scale manufacturing companies is increasing; thus, an intimate understanding of the process safety implications during laboratory synthesis and its scale-up is vital for faster and safer implementation of nanomaterials. A number of process safety incidents have occurred during scale-up, including several incidents in laboratories because of a lack of proper hazard identification.³ For example, in 2010, a graduate student was injured when trying to scale-up a conventional process involving hydrazine perchlorate derivatives synthesis from 300 mg to 10 g.⁴

One of the primary causes of safety incidents in laboratories is a lack of knowledge of safety issues arising during the synthesis route; this is especially true in cases of new materials synthesis and novel processes where the hazards of the reactions are yet to be discovered. While most universities and research laboratories rightfully require researchers to conduct a safety analysis to identify and address potential hazards, these analyses cannot be thorough if the crucial information such as heat and gas generation rate by reaction, byproduct formation, thermal stability, etc. are lacking. This crucial information is

not immediately known for novel process development in the laboratory. In addition, the guidance for completing a thorough hazard assessment is often unavailable as the scientific community only recently adopted the concepts of process safety by the means of loss prevention. As a result of these reasons, scaling up novel processes for nanomaterials synthesis can result in catastrophic incidents as hazards can compound if not done correctly. This work is intended to highlight the importance of preventive measures in order to reduce the number and severity of incidents associated with the synthesis and scale-up.

MXenes are a growing family of atomically layered two-dimensional metal carbides and nitrides with the chemical formula $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M is an early transitional metal in the periodic table, X is carbon or nitrogen, and T is a combination of several terminal groups (i.e., $-\text{F}$, $-\text{OH}$, $-\text{O}$, $-\text{Cl}$, etc.). These materials are most frequently etched from $\text{M}_{n+1}\text{AX}_n$ (MAX) phase powders, which contain post-transition groups 13–15 in the metal layer that are located between M and X layers in their crystal structure. Two-dimensional carbides can have tremendous applications as MXenes have impressive thermal and electrical conductivities. MXenes are currently being evaluated for use in lithium and nonlithium ion energy storage, fuel cell additives, supercapacitors, humidity and pH sensors, and lubricating additives.^{5,6} To date, over 60 MAX

Received: November 1, 2018

Revised: January 2, 2019

Accepted: January 4, 2019

Published: January 4, 2019

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**United States. Congress. Joint
Committee on Atomic Energy**



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Synthesis and Analysis Methods for Safety and Reliability Studies NATO Advanced Study Institute on Synthesis and Analysis, **Synthesis and Analysis Methods for Safety and Reliability Studies** G. Apostolakis, S. Garribba, G. Volta, 2012-12-06 This book originates from the NATO Advanced Study Institute on Synthesis and Analysis Methods for Safety and Reliability Studies held at Sogesta Conference Centre Urbino Italy 3-14 July 1978 The Institute co directed by Prof E J Henley and Dr G Volta was attended by 67 persons from twelve countries The focus of the Institute was on theoretical and applied aspects of reliability and risk analysis methodologies The Institute was composed of lectures workshops and guided discussions From the large quantity of written material that was used and produced during the Institute a number of papers introducing the most relevant research results and trends in the field have been selected The papers have been edited partly rewritten and rearranged in order to obtain in the end an integrated exposition of methods and techniques for reliability analysis and computation of complex systems The book is divided into four sections which correspond to fairly homogeneous areas from a methodological point of view Each section is preceded by an introduction prepared by the Editors which aims at helping the readers to put in perspective and appreciate the contribution of each paper to the subject of the section

Lees' Loss Prevention in the Process Industries Frank Lees, 2005-01-25 Over the last three decades the process industries have grown very rapidly with corresponding increases in the quantities of hazardous materials in process storage or transport Plants have become larger and are often situated in or close to densely populated areas Increased hazard of loss of life or property is continually highlighted with incidents such as Flixborough Bhopal Chernobyl Three Mile Island the Phillips 66 incident and Piper Alpha to name but a few The field of Loss Prevention is and continues to be of supreme importance to countless companies municipalities and governments around the world because of the trend for processing plants to become larger and often be situated in or close to densely populated areas thus increasing the hazard of loss of life or property This book is a detailed guidebook to defending against these and many other hazards It could without exaggeration be referred to as the bible for the process industries This is THE standard reference work for chemical and process engineering safety professionals For years it has been the most complete collection of information on the theory practice design elements equipment regulations and laws covering the field of process safety An entire library of alternative books and cross referencing systems would be needed to replace or improve upon it but everything of importance to safety professionals engineers and managers can be found in this all encompassing reference instead Frank Lees world renowned work has been fully revised and expanded by a team of leading chemical and process engineers working under the guidance of one of the world's chief experts in this field Sam Mannan is professor of chemical engineering at Texas A principles practice codes standards data and references needed by those practicing in the field **Handbook of Systems Thinking Methods** Paul M. Salmon, Neville A. Stanton, Guy H. Walker, Adam Hulme, Natassia Goode, Jason Thompson, Gemma J.M.

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Unifying Theories of Programming and Formal Engineering Methods Zhiming Liu,Jim Woodcock,Huibiao Zhu,2013-08-21 This book presents 5 tutorial lectures by leading researchers given at the ICTAC 2013 Software Engineering School on Unifying Theories of Programming and Formal Engineering Methods held in Shanghai China in August 2013 The lectures are aimed at postgraduate students researchers academics and industrial engineers They cover topics such as component based and service oriented systems real time systems hybrid systems cyber physical systems and present techniques such as inductive theorem proving model checking correction by construction through refinement and model transformations synthesis and computer algebra Two of the courses are explicitly related to Hoare and He s Unifying Theories of Programming

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Proceedings of the Third International Scientific Conference “Intelligent Information Technologies for Industry” (IITI’18) Ajith Abraham,Sergey Kovalev,Valery Tarasov,Vaclav Snasel,Andrey Sukhanov,2018-12-05 This book contains papers presented in the main track of IITI 2018 the Third International Scientific Conference on Intelligent Information Technologies for Industry held in Sochi Russia on September 17 21 The conference was jointly co organized by Rostov State Transport University Russia and V B Technical University of Ostrava Czech Republic with the participation of Russian Association for Artificial Intelligence RAAI IITI 2018 was devoted to practical models and industrial applications related to intelligent information systems It was considered as a

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