

## O 61: Focus Session: 2D Transition Metal Carbides, Nitrides and Carbonitrides I (joint session DS/MM/O)

MXenes, two-dimensional transition metal carbides, nitrides and carbonitrides, constitute one of the most rapidly growing class of 2D materials. Discovered in 2010, they have demonstrated exceptional physical, chemical, and electronic properties leading to potential applications in various fields, such as energy storage, catalysis, electromagnetic interference shielding, sensing, and biomedicine. The fundamental physical properties of MXenes are governed by their chemical composition and great research efforts are currently devoted to expanding the range of existing MXenes by tuning their stoichiometry, morphology and surface chemistry as well as adding tailored defects that can bring new functionalities. In this Focus Session, new developments related to MXene synthesis and characterization will be presented. The fundamental physical properties and interfacial processes correlated with MXene surface chemistry, defects and interlayer confinement will be discussed.

Organizers:

Dr. Tristan Petit, Head of the Young Investigator Group Nanoscale Solid-Liquid Interfaces, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin, Germany

Prof. Vincent Mauchamp, Institut Pprime, CNRS-Université de Poitiers-ISAE ENSMA, Poitiers, France

Time: Wednesday 15:00–17:00

Location: A 053

### Introduction Focus Session on MXene - V. Mauchamp & T. Petit

#### Invited Talk

O 61.1 Wed 15:15 A 053

**Sustainable synthesis of MXenes and their precursors** — ●JESUS GONZALEZ-JULIAN<sup>1</sup>, NIMA AMOUSA<sup>1</sup>, and FILIPA OLIVEIRA<sup>2</sup> — <sup>1</sup>Chair of Ceramics, Institute of Mineral Engineering (GHI), RWTH Aachen University, 52074 Aachen, Germany — <sup>2</sup>Department of Inorganic Chemistry, Faculty of Chemical Technology, University of Chemistry and Technology Prague, 166 28 Prague 6, Czech Republic

Synthesis of MXenes is typically performed using HF - or forming in situ HF through the reaction between LiF and HCl, but this chemical etching presents some limitations. HF is highly corrosive and toxic, requiring specific safety regulations, and the chemical etching is highly exothermic, limiting the amount of MXenes that can be produced in the range of grams. These two points are hindering the transfer of MXenes to real applications, despite the excellent properties of these 2D materials. Consequently, new synthesis processes are required to overcome these problems. In this work, we will present a novel sustainable synthesis route for the synthesis of the precursors (MAX phases) and the chemical etching, which is referenced as Molten Salt Shielded Synthesis or MS3. MS3 is carried out at lower temperatures than conventional synthesis routes, in air instead inert atmospheres, and does not require any milling step to obtain fine and loose powders.

O 61.2 Wed 15:45 A 053

**Tuning the molten salt etching process by in situ XRD** — ●JULIAN T. MÜLLER, ALEKSANDER GURLO, and MAGED F. BEKHEET — Technische Universität Berlin, Faculty III Process Sciences, Institute of Material Science and Technology, Chair of Advanced Ceramic Materials, Straße des 17. Juni 135, 10623 Berlin, Germany

MXenes, whose properties are mainly defined by their chemical composition and surface terminations, could be synthesized by etching an A element (A = Al, Si, etc.) from the parent MAX phase using hydrofluoric acid. This acid is of high risk to human health and leads to fluorine terminations on the surface of MXene, which are difficult to alter and may be detrimental for certain applications, e.g. electrodes in different battery systems. Etching via molten salt formation offers an alternative, less harmful way. By sophisticated selection of salts, it is possible to adjust the surface terminations and pave the way for a new generation of tailored MXenes. Gaining insight into the molten salt etching process and tuning its process parameters such as temperature, duration, and precursor ratio are keys to ensuring a qualitative MXene and saving time, energy, and cost. To support this, we are currently establishing an in situ XRD setup at the Advanced Light Source of the Lawrence Berkeley National Laboratory. It will allow measurements under various gas atmospheres up to 1450 °C and 50 bars, including a vapour phase. In the first molten salt etching experiments, we successfully gained knowledge on the etching onset, duration, and phase composition. With this measurement setup, the synthesis of MAX phases and many MXene and salt combinations await to be explored.

O 61.3 Wed 16:00 A 053

**Preparation of Magnetic MXenes by Fe intercalation** — TIM SALZMANN<sup>1</sup>, HANNA PAZNIAK<sup>2</sup>, THIERRY OUISSE<sup>2</sup>, FABRICE WILHELM<sup>3</sup>, ANDREI ROGALEV<sup>3</sup>, RALF MECKENSTOCK<sup>1</sup>, IVAN TARASOV<sup>1</sup>, MICHAEL FARLE<sup>1</sup>, and ●ULF WIEDWALD<sup>1</sup> — <sup>1</sup>University of Duisburg-Essen and Center for Nanointegration Duisburg-Essen, Germany — <sup>2</sup>Université Grenoble Alpes, CNRS, Grenoble INP, LMGP, Grenoble, France — <sup>3</sup>European Synchrotron Radiation Facility, Grenoble, France

Yet, the success of generating magnetic MXenes remains very limited since Fe, Co or Ni are incompatible with MAX phase precursors. We present an alternative approach to master magnetic properties of 2D MXenes by intercalating Fe into Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> MXene thin films on Si/SiO<sub>2</sub> in UHV conditions. Annealing of bare MXenes at T = 1023 K removes -F, -Cl and -OH. The intercalation of Fe is studied by depositing a 6 nm film on top of the MXenes and subsequent annealing. XRD shows an increase of the interplanar spacing between MXene sheets of 0.16 ± 0.02 nm. XPS and XANES reveal that Fe remains metallic and Fe diffuses 30 nm deep into the multilayers while MXenes keep intact. We study the magnetic properties by VSM and FMR. VSM suggests a new magnetic phase with M<sub>S</sub> = 660 ± 80 kA/m and a Curie temperature of 485 K while angular-dependent FMR at 9 GHz and 300 K shows two ferromagnetic and one paramagnetic signals, which we address to the remaining Fe on top and the intercalated Fe in form of quasi 2D disks and isolated Fe ions. Funded by DFG (530103526). Support by CRC/TRR 270 (405553726) is acknowledged.

O 61.4 Wed 16:15 A 053

**synthesis and characterization of 2D Mo/Ti solid solutions based MXene for hydrogen evolution reaction in alkaline media.** — LOLA LOUPIAS<sup>1</sup>, CLAUDIA MORAIS<sup>1</sup>, SOPHIE MORISSET<sup>1</sup>, CHRISTINE CANAFF<sup>1</sup>, ZHEMING LI<sup>1</sup>, PATRICK CHARTIER<sup>2</sup>, VINCENT MAUCHAMP<sup>2</sup>, THIERRY CABIOC'H<sup>2</sup>, AURÉLIEN HABIROUX<sup>1</sup>, and ●STÉPHANE CÉLÉRIER<sup>1</sup> — <sup>1</sup>Institut de Chimie des Milieux et Matériaux de Poitiers (IC2MP), Université de Poitiers, CNRS, F-86073 Poitiers, France — <sup>2</sup>Institut Pprime, UPR 3346 CNRS, Université de Poitiers, ISAE-ENSMA, BP 30179, 86962 Futuroscope-Chasseneuil Cedex, France

2D MXenes have gained an ever-increasing attention in various application fields. Their properties can be strongly tuned by modifying the M element in the Mn+1XnTx structure. Among them, Mo-based MXenes are beginning to be successfully explored in many areas. This work focuses on the synthesis and characterization of (Mo,Ti)<sub>n+1</sub>C<sub>n</sub>T<sub>x</sub> MXenes to understand their complex chemistry and to compare them with those of mono-metallic Mo<sub>2</sub>C<sub>T</sub>x and Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>. The potential of these materials as HER (hydrogen evolution reaction) catalysts is determined in alkaline medium. It is shown that Mo<sub>2</sub>Ti<sub>2</sub>C<sub>3</sub>T<sub>x</sub> MXenes are a credible alternative to Mo<sub>2</sub>C<sub>T</sub>x MXenes since the surface properties of both MXenes are similar while their composition is quite different. Indeed, (Mo,Ti)<sub>n+1</sub>C<sub>n</sub>T<sub>x</sub> require lower temperatures and shorter time for the synthesis than for Mo<sub>2</sub>C<sub>T</sub>x, a great advantage from an industrial point of view. Finally, some avenues for improving

the performance of MXenes for HER will also be described.

O 61.5 Wed 16:30 A 053

**MXenes as support for transition metal oxides electrocatalysts for water splitting application** — ●AXEL ZUBER<sup>1</sup>, ZDENĚK SOFER<sup>2</sup>, and MICHELLE BROWNE<sup>1</sup> — <sup>1</sup>CE-NESD, Helmholtz Zentrum Berlin für Materialien und Energie, Berlin (Federal Republic of Germany) — <sup>2</sup>Department of Inorganic Chemistry, University of Chemistry and Technology, Prague (Czech Republic)

To reduce the cost of energy conversion and tend towards a greener energy production, research has been trying to use first-row transition metal-based catalysts for water splitting. Despite their good activity for the oxygen evolution reaction, transition metal oxides (TMOs) performance is hindered by their low conductivity and instability under potential. To improve it, recent works have successfully combined them chemically with conductive 2D transition metal carbides and nitrides (MXenes). The rich chemistry, large surface area and conductivity of MXenes make them excellent candidates as electrocatalyst supports, but they are also keen to oxidize in water which induces a loss of these properties. In this study, different MXenes were combined with transition metal oxides and tested as electrodes for the oxygen evolution reaction. The stability of the MXene structure as well as the resulting

compound performance for the oxygen evolution reaction were investigated. The electrode materials remained stable and allowed to improve the oxygen evolution reaction overpotential of the transition metal oxide.

O 61.6 Wed 16:45 A 053

**Infrared and Raman spectroscopic analysis of functionalized graphene and Mxene layers** — ●KARSTEN HINRICHS<sup>1</sup>, MAILIS LOUNASVUORI<sup>2</sup>, FATIMA AKHTAR<sup>2</sup>, NAMRATA SHARMA<sup>2</sup>, TRISTAN PETIT<sup>2</sup>, and JÖRG RAPPICH<sup>2</sup> — <sup>1</sup>Leibniz-Institut für Analytische Wissenschaften - ISAS e.V., Application Laboratories Berlin, Schwarzschildstraße 8, 12489 Berlin, Germany — <sup>2</sup>Helmholtz-Zentrum Berlin für Materialien und Energie GmbH 14109 Berlin, Germany

Functionalized 2D-conductive materials like graphene and Mxene layers are interesting templates for catalysis but are also important for building of optical and electro-chemical sensors. Spectroscopic Raman and infrared (IR) ellipsometry analysis can access complementary information on the conductive material but also the ultrathin functional organic layer, respectively. Thereby bands due to molecular vibrations and phonons as well as free carrier absorptions are related to chemical and structural material properties. We acknowledge financial support by the European Union through EFRE 1.8/13.