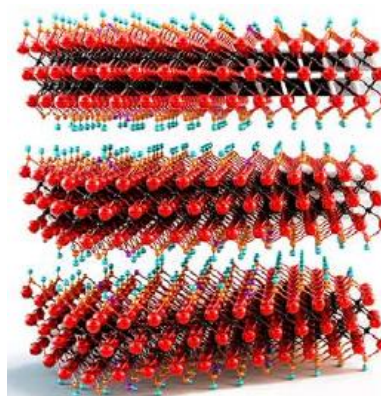


## NDnano Summer Undergraduate Research 2025 Project Summary

1. Student name & home university: Sándor Henrik Sipos, Budapest University of Technology and Economics
2. ND faculty name & department: Prof. László Forró, Dr. Bence G. Márkus, Department of Physics & Astronomy, Stavropoulos Center for Complex Quantum Matter
3. Summer project title: Tailoring the magnetic properties of MXenes
4. Briefly describe new skills you acquired during your summer research: In my summer research, I focused on doping MXenes to tailor their magnetic properties. This project introduced me to the world of two-dimensional materials, both in theory and application. To carry out the work, I learned how to handle air-sensitive compounds using a glovebox and vacuum systems. For characterization, I was trained in advanced techniques such as Vibrating Sample Magnetometry (VSM), SQUID (superconducting quantum interference device) magnetometry, Electron Spin Resonance (ESR), and Raman spectroscopy. Beyond the technical skills, I gained valuable experience collaborating within the research group and with members of collaborating research groups.
5. Briefly share a practical application/end use of your research: Tuning the properties of MXenes enables their use in a wide range of applications. For example, magnetic tuning allows integration into spintronic devices and magnetic sensors. Electrical and surface modifications can improve their performance in energy storage systems, such as supercapacitors and batteries, or enhance their catalytic activity for environmental and industrial processes.
6. 50- to 75-word abstract of your project: The aim of my project was to tune the magnetic properties of MXenes via intercalation. I selected rare-earth metal chlorides, such as  $\text{GdCl}_3$ , as dopants due to the large magnetic moment of the lanthanide ions, which can significantly influence local magnetism upon intercalation. The process was carried out in a two-zone quartz tube reactor, sealed under vacuum. Because the dopants are highly air-sensitive, all sample preparation was performed in an argon-filled glovebox. To probe the magnetic changes in the doped MXenes, I used magnetometry techniques: VSM and SQUID. Additional characterization techniques, including Raman and Electron Spin Resonance (ESR) spectroscopy, are currently ongoing. We are also planning to study the structure of the prepared materials using X-ray diffraction.
7. References for papers, posters, or presentations of your research:
  - Cahen, S., Vangelisti, R., & Bellouard, C. (2006). Structural and magnetic properties of a stage-2  $\text{HoCl}_3$ -graphite intercalation compound. *Carbon*, 44(2), 259–266.
  - Mugiraneza, S., & Hallas, A. M. (2022). Tutorial: A beginner's guide to interpreting magnetic susceptibility data with the Curie–Weiss law. *Communications Physics*, 5, 95.
  - Gogotsi, Y., & Anasori, B. (2021). The world of two-dimensional carbides and nitrides. *Science*, 372(6547), 1189–1190.

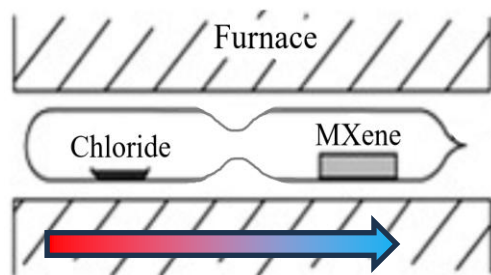
Two-dimensional materials like graphene and MXenes are at the forefront of materials research due to their exceptional electrical, magnetic, and surface properties. Their tunability makes them a promising material for applications in energy storage, sensorics, and spintronics. Current research focuses on modifying their structure and composition to unlock even more functionalities.

MXenes are a family of two-dimensional materials composed of layers held together by weak van der Waals forces. They are synthesized by selectively etching the A-element (typically Al) from MAX phases, resulting in structures with the general formula  $M_{n+1}X_nT_x$ . My research focuses on  $Ti_3C_2$  MXene, depicted in Fig. 1, which are particularly compelling due to their versatility, tunable surface chemistry, and outstanding electrochemical properties. These features make them promising candidates for applications such as supercapacitors and wearable smart electronics.



1. Figure MXene structure

The main focus of my research was to investigate and tune the magnetic properties of MXenes through chemical intercalation. I employed vapor-phase doping using a two-zone quartz tube system shown in Fig. 2. As a first step, the MXenes were heat-treated at 400 °C in high vacuum to ensure complete dehydration. Afterward, the doping setup was assembled in an argon-filled glovebox, as both the dopants and likely the intercalated products are highly air-sensitive. Rare-earth metal chlorides ( $M_{re}X_3$ ) were chosen as dopants due to the large magnetic moments of the 3+ metal ions, offering strong potential to modify the local magnetic environment within the material. Namely,

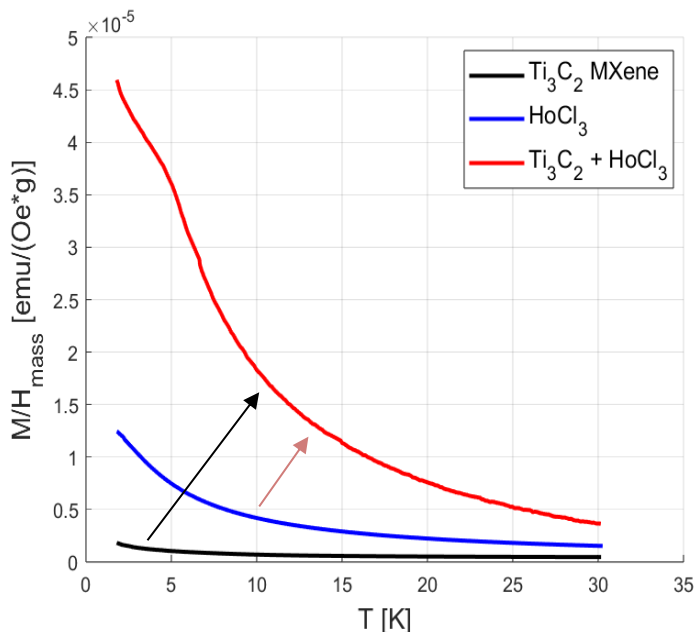


2. Figure Intercalation using the two-zone quartz tube system vapor phase technique. The arrow indicates the flow of the intercalant and denotes the temperature gradient

$GdCl_3$ ,  $TmCl_3$ ,  $TbCl_3$ ,  $HoCl_3$ , and  $NdCl_3$  were considered. The annealing process in furnaces took at least 72 hours, which resulted in vastly different shapes and sizes of doped MXenes, as well as residue inside the quartz tube, which was characteristic for the specific dopants. I chose the annealing temperature to be just below the melting points of the different dopants, to achieve maximum sublimation yields for effective intercalation. I also implemented a well-known technique used in graphite intercalation processes using additional  $AlCl_3$  as a catalyst, performing a two-step reaction chain to intercalate between the layers.

To investigate changes in the magnetic properties of the doped samples, I employed magnetization measurements. First, the samples were cooled in zero magnetic field (ZFC), and at 1.8 K a constant magnetic field was applied (typically 500 Oe) while the temperature was swept up to 300 K. This was followed by field-cooled (FC) measurements, where the samples were cooled in the presence of a magnetic field to observe differences in their magnetic response as a function of temperature. Additionally, I performed isothermal magnetization measurements at selected low temperatures by varying the external magnetic field. I started with measuring  $Ti_3C_2$  MXene first as a reference, and I found that it follows the Curie law of paramagnetism at low temperatures, with no remanent magnetization or long range ordering. I also conducted reference measurements on all the dopants themselves, and I found that some of them exhibit ferromagnetic or antiferromagnetic transitions at low temperatures, having a non-zero Curie-Weiss temperature.

Continuing the magnetometry measurements on the doped samples, the most striking results came from a  $\text{HoCl}_3$ -doped sample, whose ZFC magnetization vs. temperature curve is shown in Figure 3 together with

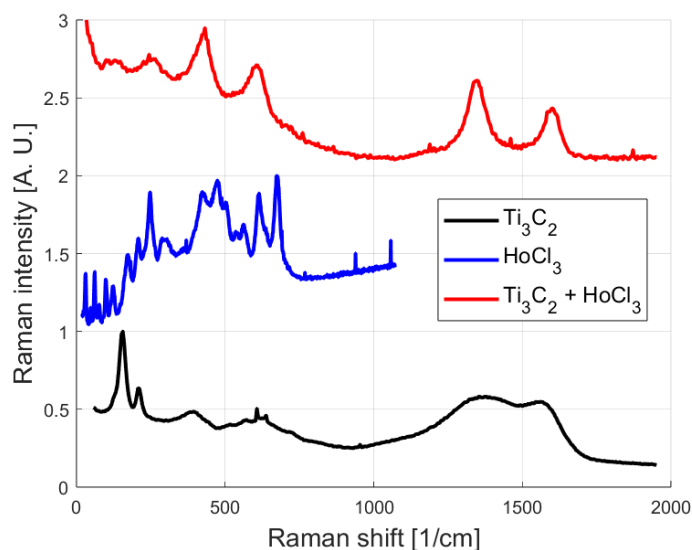


1. Figure  $\text{HoCl}_3$  doped  $\text{Ti}_3\text{C}_2$  ZFC measurement

its initial constituents. As seen in the diagram, the paramagnetic response of the MXene increased by more than an order of magnitude in the Curie–Weiss regime, and a magnetic ordering transition appears around 5 K, indicated by a kink in the magnetization curve. Other doped samples, such as those with  $\text{GdCl}_3$ ,  $\text{TmCl}_3$ , and  $\text{TbCl}_3$ , also exhibited strong paramagnetic enhancement. Several additional samples are prepared, and a key next step in the project is to systematically measure their magnetic properties.

I have also utilized Raman spectroscopy to further study the changes in MXenes through doping process. This measurement method detects inelastic light scattering which is sensitive to the vibration of molecules and crystal lattices. This technique provides insight into chemical composition, bonding and structural changes in the materials. I also started my Raman measurement with references as MXene itself and the dopants. The same batch of  $\text{HoCl}_3$ -doped MXene sample was investigated with Raman spectroscopy is shown in Figure 4. Here, the doped sample has peaks in the low-wavenumber regime ( $300\text{--}800\text{ cm}^{-1}$ ) which cannot be connected to modes in the pristine MXene. In the high-wavenumber regime ( $1200\text{--}1800\text{ cm}^{-1}$ ) the peaks get more emphasized and distinct which can be attributed to the carbon atoms being more exposed. These new peaks and peak shifts could be a sign of doping/intercalation, but these samples require a deeper analysis.

The next steps in the research is to measure X-ray diffraction on the doped samples to verify what kind of crystal phases are present, to ultimately verify the success of doping. I am also currently working on electron spin resonance (ESR) spectroscopy to study the local magnetic and electric properties of these materials.



4. Figure  $\text{HoCl}_3$  doped  $\text{Ti}_3\text{C}_2$  Raman spectrum