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Atomic-resolution in-situ cooling study of functionally terminated 2D transition metal carbides

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MXenes are a family of two-dimensional materials synthesized by etching select elements from a parent MAX crystal. Aided by their ability to undergo surface group transformations, MXenes offer researchers a larger design space when compared to other families of 2D materials such as graphene and transition-metal dichalcogenides [1]. In fact, recent work has shown that modifying surface groups can lead to giant (18%) lattice expansions, as in the case of Ti3C2Te, and superconductivity in bare (no surface group) Niobium MXenes[2].

In 2020, Kamysbayev et al. introduced a general method for installing and removing surface groups by using Lewis molten salts. Figure 1 outlines this procedure and shows atomic resolved images as well as nanoscale XEDS line scans confirming the synthesis of each MXenes. In this contribution, we expand upon this work by characterizing the chemical composition and structure of novel MXenes synthesized with organic amine surface groups.

Like other organic materials, the organic surface groups for these MXenes are not stable under the electron beam conditions needed for atomic-resolution imaging and spectroscopy at room temperature. Borrowing elements from recent advancements in imaging electron-dose sensitive samples using Cryo-STEM , we plan to increase the sample robustness by cooling the MXenes to liquid nitrogen temperatures [3].

In order to cool the MXene sheets, we will use a Gatan cold stage capable of reaching -170°C. The characterization will be conducted utilizing an aberration-corrected cold field emission JEOL ARM200CF operated at 200kV primary electron energy. With the emission current set to 15μA, the electron probe will be operated at 24mrad convergence semi-angle. High angle annular dark field, low angle annular dark field and annular bright field detectors will be set to inner angles of 75 mrad, 30 mrad, 11mrad. XEDS measurements will be conducted using a large solid-angle Oxford XMAX100TLE detector. The ARM200CF is equipped with a post-column Gatan Continuum GIF spectrometer providing capabilities for EELS measurements. In this contribution, we will report atomic-resolved XEDS and EELS maps of beam sensitive MXenes. Additionally, we will report atomic resolved images of functional groups such as organic amine and diamine groups. From these images we will characterize the effects of surface groups on the MXene lattice and the interatomic spacing of the MXene nanosheets. [4]

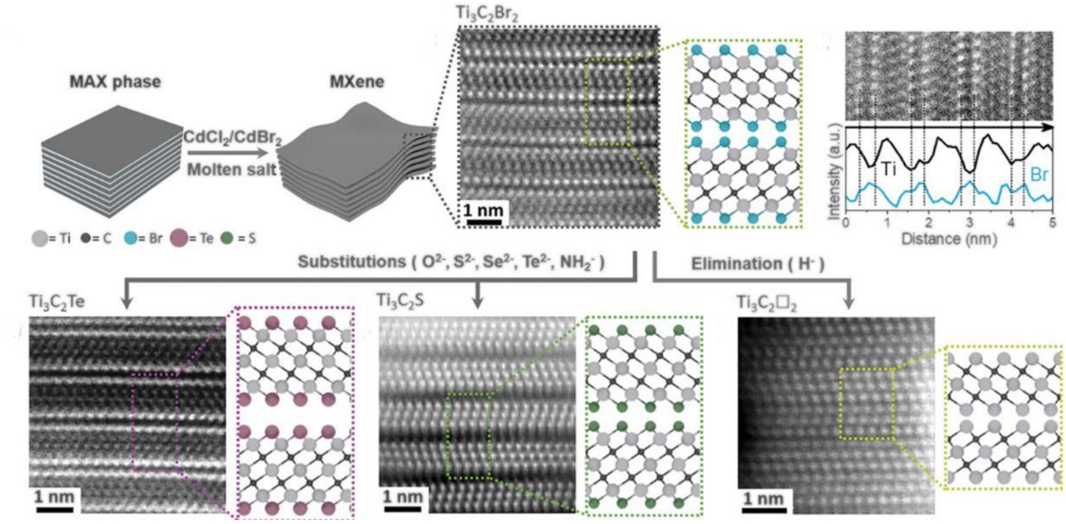

Figure 1. General strategy for installing and removing surface groups in MXenes using molten salts. [2]

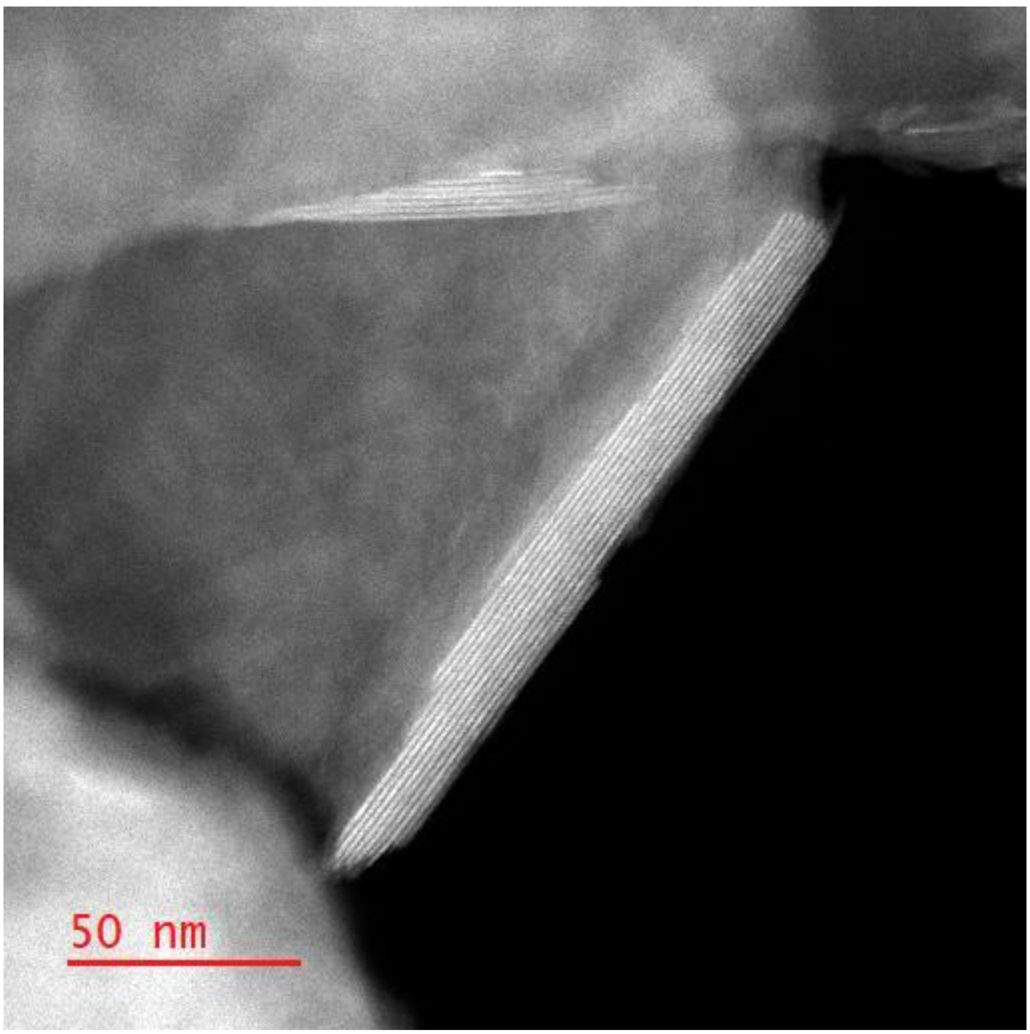

Figure 2. Figure 2 shows functionalized MXene sheets with the electron beam parallel to the (0001) axis.

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