

Supporting Information

On the Nature of Terminating Hydroxyl Groups and Intercalating Water in $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes: A Study by ^1H Solid-State NMR and DFT Calculations

*Takeshi Kobayashi,^a Yangyunli Sun,^b Kaitlyn Prenger,^c De-en Jiang,^b Michael Naguib,^c Marek
Pruski^{a,d,*}*

^a Ames Laboratory, U.S. Department of Energy, Ames, IA 50011, United States

^b Department of Chemistry, University of California, Riverside, CA 92521, United States

^c Department of Physics and Engineering Physics, Tulane University, New Orleans, LA 70118,
United States

^d Department of Chemistry, Iowa State University, Ames, IA 50011, United States

Synthesis of MAX phase Ti_3AlC_2

MAX Ti_3AlC_2 was synthesized similarly to that reported by Huang and Mochalin.¹ Powders of titanium (Alfa Aesar, 99.5%, -325 mesh), aluminum (Alfa Aesar, 99.5%, -325 mesh), and graphite (Alfa Aesar, 99%, APS 7-11 μm) were mixed in ratios of 3:1.2:1.88 in a Turbula T2F mixer for 3 h, then furnace in a tube furnace under continuous flow of argon at 1600 °C for 2 h with a heating rate of 10°C/minute. After cooling, powders were ground to -325 mesh for use in etching.

^1H - ^1H SQ/SQ correlation spectra of MXene annealed at 110 °C.

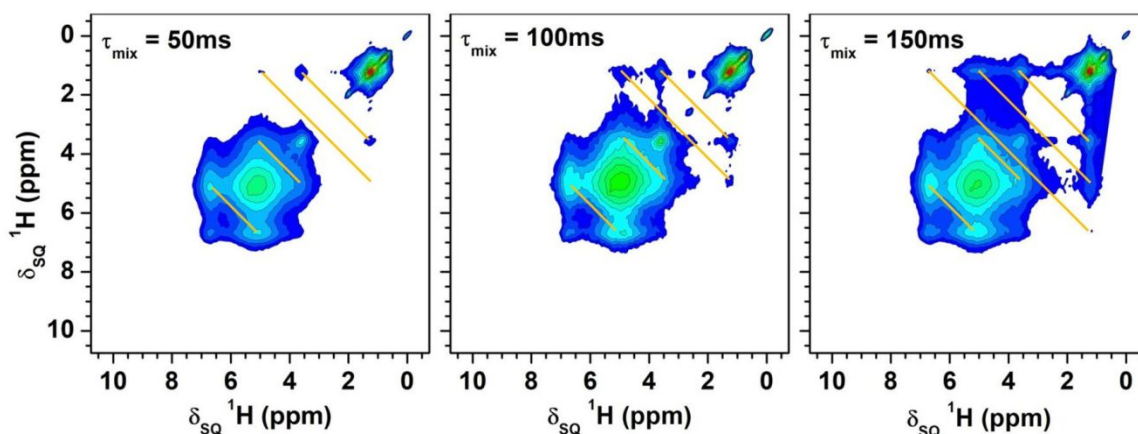


Figure S1. 2D ^1H - ^1H SQ/SQ spectra of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene annealed at 110 °C. and 200 °C, taken with $\tau_{\text{mix}} = 50$ ms, 100 ms, and 150 ms, 256 t_1 points, and $\Delta t_1 = 55.6 \mu\text{s}$.

DFT calculations

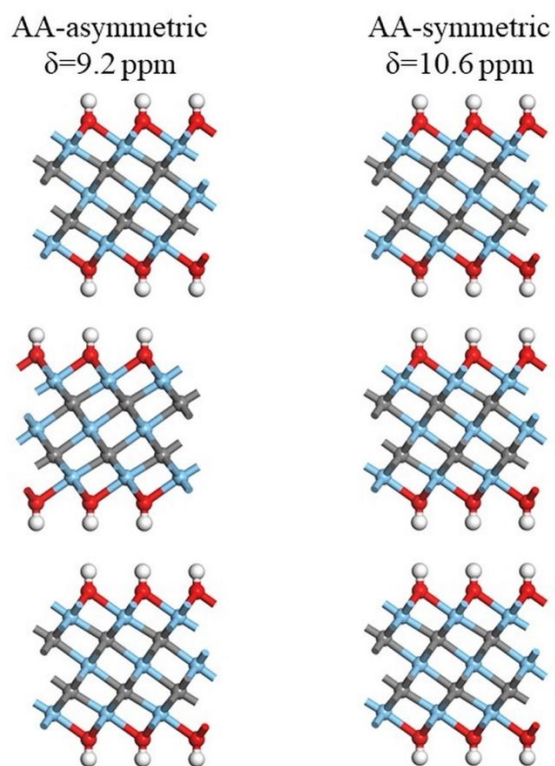


Figure S2. Computed ^1H chemical shifts for Ti–OH terminations on AA-stacking MXene.

References

- (1) Huang, S.; Mochalin, V. N. “Hydrolysis of 2D Transition-Metal Carbides (MXenes) in Colloidal Solutions”, *Inorg. Chem.* **2019**, 58, 1958–1966 .