In Situ High-Pressure X-ray Diffraction and Raman Spectroscopy Study of Ti$_3$C$_2$T$_x$ MXene

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Abstract

The lattice stability and phonon response of Ti$_3$C$_2$T$_x$ MXene at high pressure are important for understanding its mechanical and thermal properties fully. Here, we use in situ high hydrostatic pressure X-ray diffraction (XRD) and Raman spectroscopy to study the lattice deformation and phonon behavior of Ti$_3$C$_2$T$_x$ MXene. XRD spectra indicate that no phase transformation occurs up to the pressure of 26.7 GPa. The elastic constant along a lattice parameter was calculated to be 378 GPa. In the Raman spectra obtained at high-pressure, the out-of-plane phonon modes (A$_{1g}$ at ~ 210, ~ 504, and ~ 711 cm$^{-1}$) exhibit monotonic blueshifts with increasing pressure. The Grüneisen parameters of these three modes were calculated to be 1.08, 1.16, and 0.29, respectively. These results enrich the basic property data of Ti$_3$C$_2$T$_x$ MXene and would benefit the further understanding of this novel material.

Keywords: Ti$_3$C$_2$T$_x$ MXene, High-pressure XRD, High-pressure Raman, Grüneisen parameter

Background

After the intensive studies of graphene [1] and transition metal dichalcogenides (TMDs) [2–5] for a decade, two-dimensional (2D) metal carbides (MXenes) have been drawing much attention recently owing to their extraordinary electrical properties [6, 7]. The Ti$_3$C$_2$ MXene is a layered material with Van der Waals stacked structure, in which each layer contains two carbon atom planes sandwiched among three Ti atom planes. In the energy band structure of Ti$_3$C$_2$ MXene, the conduction band touches the valence band at the Γ point, which indicates that Ti$_3$C$_2$ MXene is a half-metallic material [8]. More interestingly, the band structure of Ti$_3$C$_2$ MXene can be opened up slightly by the surface functional groups (which are annotated as “T” in the formula) [8], such as –F, –O, and –OH [9]. These functional groups are generated in the solution etching of MnAlC$_n$+1 phase [9], which then forms a Ti$_3$C$_2$T$_x$ MXene with tunable electrical properties. The electrical conductivity of Ti$_3$C$_2$T$_x$ MXene was measured to be 4.2 × 10$^{-4}$S/m [10], which is superior to most TMDs. To date, Ti$_3$C$_2$T$_x$ MXenes have shown potential applications in advanced supercapacitors [11], Li-batteries [12], electromagnetic shielding [10], antibacterial [13], and light emission [14].

In addition to high electrical conductivity, the elastic property of Ti$_3$C$_2$T$_x$ MXene also attracts much attention. Theoretical calculations predicted that this ultrathin carbide has a Young’s modulus of as high as ~ 500 GPa [15–17]. Lipatov et al. recently used nanoindentation to determine that the Young’s modulus of monolayer Ti$_3$C$_2$T$_x$ MXene was 330 Gpa [18]. These values are much higher than those of MoS$_2$ [18] and are comparable to those of monolayer graphene [19]. Recently, Ghidiu et al. measured the high-pressure X-ray diffraction (XRD) spectra of Ti$_3$C$_2$T$_x$ MXene up to 3 GPa and observed no phase transformation [20]. However, as the pressure loaded in ref. [20] was too low, the phase stability and lattice deformation of Ti$_3$C$_2$T$_x$ at higher pressure are still unknown.

Raman spectroscopy acts as a useful non-destructive tool to investigate the crystal structure and phonon vibration of 2D materials such as graphene [21] and TMDs [2]. The composition of Ti$_2$CT$_x$ [22] and phase stability of Ti$_3$C$_2$T$_x$ MXene at different annealing conditions [10] can be probed by using confocal Raman
measurements. Recently, the phonon dispersion of \(\text{Ti}_3\text{C}_2\text{T}_x\) MXene was theoretically calculated by Hu et al. [23, 24], thus enabling a further understanding of the Raman spectra of this material. However, the high-pressure Raman spectroscopy of \(\text{Ti}_3\text{C}_2\text{T}_x\) is still lacking. Moreover, the phonon response of \(\text{Ti}_3\text{C}_2\text{T}_x\) as a function of pressure is unknown.

In this paper, we prepared \(\text{Ti}_3\text{C}_2\text{T}_x\) thin flakes and measured their pressure-dependent XRD and Raman spectra up to 26.7 GPa. The elastic constants of \(\text{Ti}_3\text{C}_2\text{T}_x\) were calculated from XRD diffraction peak shifts by the Murnaghan equation. The positive Grüneisen parameters of out-of-plane phonons were obtained from their pressure-dependent Raman shift and lattice parameter deformation ratio. The obtained results would benefit the further understanding of the mechanical and phonon-vibrational behavior of \(\text{Ti}_3\text{C}_2\text{T}_x\) MXene.

**Results and Discussions**

Before conducting high-pressure measurements, we first investigated the basic material properties of the exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes. An optical image of the exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes deposited on Si/SiO\(_2\) (300 nm) substrate is shown in Fig. 1a. Light green contrast can be observed for the exfoliated flakes. As reported by Miranda et al., the optical contrast of \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes depends on the flake thickness strongly, where thicker flakes always show higher contrast, while thin flakes exhibit low contrast [25]. The light green contrast of most of the flakes in Fig. 1b indicates their thin thickness. An atomic force microscope (AFM) topographic image of exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes is shown in Fig. 1b. The flakes in the mapping area show surfaces with high roughness, which is typical for \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes [26]. The thickness of a typical thin flake can be determined from its line profile (inset of Fig. 1b) across the marked position in Fig. 1b to be 170 nm. A scanning electron microscope (SEM) image of an exfoliated flake is shown in Fig. 1c. The laminated structure of \(\text{Ti}_3\text{C}_2\text{T}_x\) can be seen clearly, indicating the successful preparation of \(\text{Ti}_3\text{C}_2\text{T}_x\) layered samples [10].

We further measured the XRD spectra of raw \(\text{Ti}_3\text{C}_2\text{T}_x\) powder, as shown in Fig. 1d. This XRD pattern is an analogy to previous reports [10]; thus, the prominent peaks at 8.95°, 18.28°, and 27.7° can be assigned to the diffractions of (002), (004), and (006) planes. Compared to the prominent peaks, the intensity of diffraction peaks of minor phases (anatase TiO\(_2\)(101) at 25.3°, JCPDS Card No. 71-1116) is relatively weak, indicating the high purity of \(\text{Ti}_3\text{C}_2\text{T}_x\) phase in the obtained powder. The (002) peak emerges at an angle slightly lower than that reported by Han et al. (9.21°) [10]. The calculated \(c\) lattice parameter, 19.66 Å, is larger than

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**Fig. 1**

- **a** Optical image of ultrasonically exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes.
- **b** AFM topographic image of ultrasonically exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes, and a line profile across the marked dashed line is shown as an inset, indicating the \(\text{Ti}_3\text{C}_2\text{T}_x\) flake thickness of 170 nm.
- **c** SEM image of ultrasonically exfoliated \(\text{Ti}_3\text{C}_2\text{T}_x\) flakes.
- **d** XRD spectra of \(\text{Ti}_3\text{C}_2\text{T}_x\) raw powder.
the reported value (19.2 Å) [10]. It should be noted that since the interlayer space may be tuned by different densities of chemical groups and ions, such as –F, –OH, and Li+, the c lattice parameter varies substantially from 19.2 Å to 58.8 Å in different studies [10, 20, 26, 27]. The c lattice parameter for our sample is very close to the low value that was measured for Ti3C2Tx powder simply using HF as etchant [10]. The XRD spectra of Ti3C2Tx flakes measured at different pressures until 26.7 GPa are shown in Fig. 2a. It can be seen that the spectra measured at different pressures are similar to each other, while no new diffraction peak can be found. This finding indicates no phase transformation occurs with the pressure up to 26.7 GPa. In Fig. 2a, all the diffraction peaks shift to large angles with increasing pressure, indicating the shrinkage of the Ti3C2Tx lattice. Such pseudo-negative compressibility has also been observed for Ti3C2Tx [20] and other low dimensional materials with a layered structure, such as graphite [28], graphene oxide [29, 30], MoS2 [31], clay [32], and titanates [33]. The (002) peak shifts from 2.883° to 3.162° as the pressure increased from 1.8 GPa to 26.7 GPa. The deformation ratio of lattice parameter c, c/c0, as a function of pressure, can be calculated from the shift of (002) peak. Moreover, the deformation ratio of a, a/a0, can be calculated from the shift of (110) peak. As shown in Fig. 2b, the lattice parameters c and a are deformed by 9.1% and 2.4%, respectively, at a pressure of 26.7 GPa. In the low-pressure region at ~ 3 GPa, the compressive ratio of lattice parameter c is 3%. In the previous high-pressure XRD measurement of Ti3C2Tx flakes, a slightly larger c compressive ratio of 4% for dry Ti3C2Tx flakes was reported by Ghidiu et al. [11](Fig. 2b). This difference could be induced by a larger lattice parameter c(25.1 Å) of the sample used by Ghidiu et al. [11] with respect to that of ours(19.66 Å).

To obtain the elastic constants, the c and a compressive ratios in Fig. 2b are further fitted by using the Murnaghan equation [34].

$$r(P)/r_0 = \left[\left(\frac{\beta}{\beta_0}\right)P + 1\right]^{-1/\beta}$$  (1)

where r represents the lattice constants along c and a axes, $\beta_0$ is the linear compressibility, and $\beta$ is the pressure derivative of $\beta$.

The fitted r/r0 curves of a and c are plotted as solid lines in Fig. 2b. It can be seen that the experimental results fit the equation expectation very well. The best fitting generates $\beta_0$ and $\beta$ for c as 67.7 GPa and 25.5, respectively. While for lattice parameter a, $\beta_0$ and $\beta$ are calculated to be 387.4 GPa and 72.1, respectively (Table 1). For ultrathin 2D materials such as graphene, Young’s modulus (1TPa) is very close to the $\beta_0$ of thick graphite [19, 28]. Therefore, $\beta_0$ can be used as a substitution to evaluate the elastic constant of Ti3C2Tx. Young’s modulus of Ti3C2Tx was recently measured by Lipatov et al. to be 330 GPa [18], which is consistent with the $\beta_0$ in our study. Our measured values are also comparable to the elastic constants of Ti3C2 that were calculated in other studies [15, 17] (Table 1). The $\beta_0$ at c axis is larger than that of graphite ($\beta_0$ = 35.7 GPa), while the $\beta_0$ at a axis is smaller than that of graphite ($\beta_0$ = 1250 GPa) [28]. The $\beta_0$ of Ti3C2Tx is higher than the bulk modulus of MoS2 (270 Pa) [35] and is also comparable to that of graphene oxide (210 GPa) [36], indicating a high elastic constant of Ti3C2Tx Mxene among 2D materials.
High-pressure Raman spectra of Ti$_3$C$_2$T$_x$ samples were measured at different compressive pressures up to 25.5 GPa, as shown in Fig. 3a. The Raman spectra obtained at different decompressive pressures are shown in Fig. 3b. At low compressive pressures, Ti$_3$C$_2$T$_x$ Mxene exhibits three major Raman bands at ~ 210, ~ 500, and 700 cm$^{-1}$. It should be noted that the Raman spectra of Ti$_3$C$_2$T$_x$ MXene vary significantly in different kinds of literature. Hu et al. [23] reported strong Raman peaks at ~ 200 cm$^{-1}$ and 720 cm$^{-1}$, while other bands at 400 cm$^{-1}$ were quite broad. However, Han et al. [10] and Zhu et al. [37] observed a sharp peak at ~ 200 cm$^{-1}$, but other bands were all broad. Xue et al. [14] only observed broad peaks from 100 to 700 cm$^{-1}$. The Raman spectra in Fig. 3 are different from those in ref. [10, 14, 23, 37]. This difference could be induced by different types and concentrations of chemical groups on Ti$_3$C$_2$T$_x$ MXene. Further interpretation of these different Raman bands needs to recall the phonon dispersion of Ti$_3$C$_2$T$_x$ that was theoretically calculated by Hu et al. [23, 24]. The space group of Ti$_3$C$_2$T$_x$ was P6$_3$/mmc [23]. The number of atoms ($N$) in a primitive cell of Ti$_3$C$_2$T$_x$ was calculated to be 7, 7, and 9 for $T = $ O, F, and $-OH$, respectively, given $x = 2$. At $\Gamma$ point of the first Brillouin zone, the following optical phonons are predicted to be existing for different Ti$_3$C$_2$T$_x$ MXenes: $\Gamma_{\text{optical}} (\text{Ti}_3\text{C}_2\text{O}_2) = 6E_g + 3A_{1g}$, $\Gamma_{\text{optical}} (\text{Ti}_3\text{C}_2\text{F}_2, \text{Ti}_3\text{C}_2(\text{OH})_2) = 8E_g + 4A_{1g}$ [23]. The atom vibrations of different Raman active modes of Ti$_3$C$_2$F$_2$ and Ti$_3$C$_2$(OH)$_2$ are illustrated schematically in Table 2. Their frequencies were theoretically calculated by Hu et al. [23] and are listed in Table 2. For Ti$_3$C$_2$(OH)$_2$, there are four out-of-plane modes ($A_{1g}$: 218, 514, 684, and 3734 cm$^{-1}$) and four in-plane modes ($E_g$: 138, 278, 437, and 622 cm$^{-1}$). For Ti$_3$C$_2$F$_2$, there are three $A_{1g}$ modes (190, 465, and 694 cm$^{-1}$) and three $E_g$ modes (128, 231, and 612 cm$^{-1}$) [23]. However, it can be seen the calculated phonon frequencies of pure Ti$_3$C$_2$F$_2$ or Ti$_3$C$_2$(OH)$_2$ cannot fit the experimental Raman spectra of Ti$_3$C$_2$T$_x$ in Fig. 3. Because the surface of Ti$_3$C$_2$T$_x$ is usually attached by a different type of chemical groups, a full interpretation of the experimental Raman spectra in Fig. 3 needs to consider the hybridized vibration modes of $-F$ and $-OH$ [23]. In a previous research [23], the Raman bands at ~ 200, ~ 500, and ~ 700 cm$^{-1}$ were assigned to $\omega_2$, $\omega_6$, and $\omega_3$, respectively. Following this instruction, the prominent Raman bands at 205.6, 490.2, and 702.5 cm$^{-1}$ of Fig. 3a, b can be assigned to $\omega_2$, $\omega_6$, and $\omega_3$ modes, respectively. Interestingly, these modes are all out-of-plane modes. Other Raman modes are difficult to be isolated from their neighboring modes due to

| Mode          | $\beta_0$ (cm$^{-1}$) | $\beta$ |
|---------------|------------------------|---------|
| c axis        | 67.7                   | 25.5    |
| a axis        | 387.4                  | 72.1    |
| 502 (Young’s modulus) | Borysiuk et al. [16] |
| 473 (C$_1$)   | Bing et al. [17]       |
| 491 (C$_1$)   | Bai et al. [15]        |
| 447 (Young’s modulus) | Ning et al. [17] |
| 330 (Young’s modulus) | Lipatov et al. [18] |

Fig. 3a Raman spectra of Ti$_3$C$_2$T$_x$ flakes at different compression pressures; b Raman spectra obtained at different decompression pressures. Note that the unit of pressures in a and b is GPa
their low intensities. To obtain robust data by eliminating the uncertainties, only these three modes are considered in the following calculations and analyses.

In Fig. 3a, b, it can also be seen that the relative intensities of these in-plane modes increase with the increasing compressive pressures (Fig. 3a). When the compressive pressure is ≥ 12.6 GPa, a new peak at ~ 600 cm⁻¹ (ω₄) emerges and becomes the prominent peak. In the decompression process, the intensities of this ω₄ mode decrease significantly. The Raman spectra obtained at 0 GPa of decompression pressure contain almost all the in-plane and out-of-plane phonon modes. Such emergence of in-plane modes at high compressive pressure might be related with flake fracture or orientation rotation-induced polarization. Research about this effect is still ongoing and will be reported in the future.

With increasing pressure from 0.8 GPa to 25.6 GPa, ω₂, ω₃, and ω₄ all show monotonic increasing blueshifts (Fig. 4a–d), which are similar to the pressure-dependent blueshifts of graphite [28] and MoS₂ [31]. At 25.6 GPa, these three modes' blueshifts are 66.7, 85.1, and 60 cm⁻¹, respectively. Such pressure-dependent blueshifts are much larger than those of MoS₂ [31]. To quantify the Raman shift vs pressure, the Raman shift

Table 2 Assignment of typical Raman peaks in this work and reference. The theoretically calculated phonon energies of Ti₃C₂(OH)₂ and Ti₃C₂F₂ are listed for comparison. The vibration directions of atoms in a unit cell are also shown schematically. The unit of Raman shift is cm⁻¹.

| Mode     | This work | Ref [10, 23] | Ti₃C₂(OH)₂ [23] | Ti₃C₂F₂ [23] |
|----------|-----------|--------------|-----------------|--------------|
| ω₁ (E₂)  | 205.6     | 210          | 138             | 128          |
| ω₂ (A₁g) | 702.5     | 730          | 218             | 190          |
| ω₃ (A₁g) | 630       | 622          | 684             | 604          |
| ω₄ (E₂)  |           | 278          | 622             | 621          |
| ω₅ (E₂)  | 490.2     | 514          | 514             | 465          |
| ω₆ (A₁g) |           | 437          |                 |              |

Fig. 4 Raman shifts of different phonon modes as a function of different compressive pressures (solid sphere) and decompressive pressures (opened circles): a 210 cm⁻¹, b 504 cm⁻¹, c 620 cm⁻¹, and d 711 cm⁻¹. Solid lines are the fitting results using the equation ω(P)/ω₀ = [(δ₀/δ₀)P + 1]^{δ/2}.
plots in Fig. 4a, b, d were fitted using the following equation [28]:

\[ \frac{\omega(P)}{\omega_0} = \left[ (\delta_0/\delta') P + 1 \right]^{\delta'} \]  

(2)

where \( \delta_0 \) and \( \delta' \) are the logarithmic pressure derivative \( (d\ln\omega/dP)_{P=0} \) and the pressure derivative of \( d\ln\omega/dP \), respectively. The fitted results are plotted as solid lines in Fig. 4a, b, d. Due to the high uncertainties at the low-pressure region, the Raman mode at 620 cm\(^{-1}\) was not fitted. It can be seen in Fig. 4a, b, d that the fitted curves are consistent with the experimental results, indicating the high accuracy of the fitting process. The obtained parameters of \( \delta_0 \) and \( \delta' \) are listed in Table 3.

For anisotropic 2D materials with an atomic thickness, such as MXene and graphite, two independent components of the Grüneisen tensors are usually associated with the strains that are parallel and perpendicular to the c axis. For simplicity, we adopted the scaling relationship proposed by Zallen et al. [38], which has been used to fit the pressure-dependent Raman shift of graphite by Hanfland et al. [28]

\[ \omega(P)/\omega_0 = \left[ r(P)/r_0 \right]^{3\gamma} \]  

(3)

where \( r \) refers to the in-plane and out-of-plane lattice constants for intralayer and interlayer modes, respectively. \( \gamma \) is equivalent to the Grüneisen parameter that was defined in other studies [39, 40].

As only the out-of-plane modes are observed in the compression process, \( c \) lattice parameter as a function of hydrostatic pressure is adequate for calculation. We adopted the (002) plane’s space distance data of 0 to 26.7 GPa in Fig. 2b for the calculation of Grüneisen parameter \( \gamma \). The averaged \( \gamma \) up to 26.7 GPa for \( \omega_2 \), \( \omega_5 \), and \( \omega_3 \) were calculated to be 1.08, 1.16, and 0.29, respectively (Table 2). Similar to the graphite, the smaller \( \gamma \) of \( \omega_3 \) compared with the other two modes indicates a smaller change in force constants is involved in the rigid-layer motion [28]. To the best of our knowledge, the Grüneisen parameters of Ti\(_3\)C\(_2\)\(_x\) have not been reported yet. However, we can still compare our data with other 2D materials. Zha et al. reported Grüneisen parameters as 4–5 for acoustic phonon modes of Ti\(_2\)CO\(_2\) [41]. Because the Grüneisen parameters of high-frequency optical modes are usually one or two orders lower than those of the low-frequency modes [28], the Grüneisen parameters for optical phonons of Ti\(_2\)CO\(_2\) can be estimated to be 0.05–0.5, which are similar to those of our values for Ti\(_3\)C\(_2\)\(_x\). Recently, Peng et al. [42] reported the Grüneisen parameters at room temperature as 1.22, 1.20, and 1.15 for MoS\(_2\), MoSe\(_2\), and WS\(_2\), respectively, which are larger than our results. Our results are also smaller than those of graphene (1.99 for \( E_{2g} \) mode) [40] and graphite (1.06 for \( E_{2g} \) mode) [28]. This finding indicates that Ti\(_3\)C\(_2\)\(_x\) MXene has the weakest bonding anharmonicity among these ultrathin 2D materials [42].

**Conclusions**

In conclusion, we measured lattice deformation and phonon response of Ti\(_3\)C\(_2\)\(_x\) thin flakes at different hydrostatic pressures up to 26.7 GPa. No phase transformation has been observed below a pressure of 26.7 GPa. All the phonon modes show a positive frequency shift with increasing pressures. The positive Grüneisen parameters of three out-of-plane phonons are calculated to be 1.08, 1.16, and 0.29. Our results increase understanding of the mechanical and thermal properties of Ti\(_3\)C\(_2\)\(_x\) at high pressures.

**Methods**

Ti\(_3\)C\(_2\)\(_x\) powder was prepared by a method reported by reference [43]. Briefly, Ti\(_3\)AlC\(_2\) powder (Forsman, 10 g) was etched by HF solution (160 ml) at room temperature for 5 h. The obtained Ti\(_3\)C\(_2\)\(_x\) powder was dispersed into DI water and ultrasonically exfoliated at a power of 700 W. The resulted solution was separated after being stored by 24 h. The obtained upper layer solution was used for further Raman, atomic force microscope (AFM), and scanning electron microscope (SEM) analyses. X-ray diffraction (XRD) spectra at ambient pressure were measured using an X-ray diffractometer (Rigaku, MiniFlex600). SEM images were obtained using a scanning electron microscope (Hitachi, Su1510). In situ high-pressure XRD measurements were performed at the Shanghai Synchrotron Radiation Facility by a gasket high-pressure diamond anvil cell (DAC) at room temperature. To produce a hydrostatic environment around the sample, we used methanol/ethanol/water (16:3:1) as a pressure transmitting medium. The pressure was determined by the pressure-dependent spectral shift of the sharp ruby fluorescence R1 line. The sample was placed in a stainless steel gasket hole (100 μm in diameter) with a diamond culet (400 μm in diameter). High-pressure Raman scattering measurements were performed using a Renishaw inVia Raman spectrometer with an

| Mode  | \( \omega_0 \) (cm\(^{-1}\)) | \( \delta_0 \) (GPa\(^{-1}\)) | \( \delta' \) | \( \gamma \) |
|-------|----------------------------|-----------------|-------------|-------|
| \( \omega_2 \) | 210 | 0.025 | 0.21 | 1.08 |
| \( \omega_5 \) | 504 | 0.028 | 0.079 | 1.16 |
| \( \omega_3 \) | 711 | 0.009 | 0.056 | 0.29 |

Table 3 Calculated pressure-dependent parameters of Ti\(_3\)C\(_2\)\(_x\). \( \gamma \) refers to the averaged Grüneisen parameters that were calculated using equation \( \gamma = -d\ln\omega/3dlnP \).
excitation wavelength of 532 nm. The topographical measurements were conducted on an AFM instrument (Bruker, Innova).

The fitting of diffraction peak positions and Raman peak shifts was conducted on OriginPro package. A user-defined function, \( y = (A1 \times x + 1)^{A2} \), was used by setting A1 and A2 as the fitting parameters. Proper fitting can be obtained easily by a simplex method.

**Abbreviations**

2D: Two dimensional; AFM: Atomic force microscope; DAC: Diamond anvil cell; SEM: Scanning electron microscope; TMDs: Transition metal dichalcogenides; XRD: X-ray diffraction

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**Availability of Data and Materials**

We declare that the materials used in this manuscript, including all relevant raw data, will be freely available to any scientist wishing to use for non-commercial purposes, without breaching participant confidentiality.

**Authors’ Contributions**

This work is carried out by the collaboration between all the authors. LZ, WS, and LF synthesized the Ti\(_3\)C\(_2\)Tx ultrathin flakes and conducted the sample characterization and the data analysis. YH conducted the high-pressure Raman and XRD measurements. KS and XH conducted the XRD data analysis. JY and CL prepared the raw Ti\(_3\)C\(_2\)Tx samples. All authors read and approved the final manuscript.

**Competing Interests**

The authors declare that they have no competing interests.

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