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# Single- and few-layer WTe<sub>2</sub> and their suspended nanostructures: Raman signatures and nanomechanical resonances

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Single crystal tungsten ditelluride (WTe<sub>2</sub>) has recently been discovered to exhibit non-saturating extreme magnetoresistance in bulk; it has also emerged as a new layered material from which atomic layer crystals can be extracted. While atomically thin WTe<sub>2</sub> is attractive for its unique properties, little research has been conducted on single- and few-layer WTe2. Here we report the isolation of single- and few-layer WTe2, as well as the fabrication and characterization of the first WTe<sub>2</sub> suspended nanostructures. We have observed new Raman signatures of single- and few-layer WTe2 that have been theoretically predicted but have not been reported to date, in both on-substrate and suspended WTe<sub>2</sub> flakes. We have further probed the nanomechanical properties of suspended WTe<sub>2</sub> structures by measuring their flexural resonances, and obtain a Young's modulus of  $E_{\rm Y} \approx 80$  GPa for the suspended WTe<sub>2</sub> flakes. This study paves the way for future investigations and utilizations of the multiple new Raman fingerprints of single- and few-layer WTe<sub>2</sub>, and for explorations of mechanical control of WTe<sub>2</sub> atomic layers.

# Introduction

Following the advent of graphene,<sup>1</sup> atomic layer two-dimensional (2D) crystals derived from layered materials, especially transition metal di-chalcogenides (TMDCs) such as MoS<sub>2</sub>,<sup>2</sup> WSe<sub>2</sub><sup>3</sup> and MoTe<sub>2</sub>,<sup>4</sup> have generated enormous interest. The attractive new properties of these 2D crystals have spurred remarkable efforts on exploring new electrical,<sup>5</sup> optical,<sup>6</sup> and mechanical devices<sup>7</sup> based on these layered materials. Among various device structures, suspended, movable 2D flakes make a special platform with controllable mechanical degrees of freedom, which not only frees the crystal from the bonding of substrates, but can also enhance device performance and versatility. For example, suspended graphene transistors exhibit ultra-high mobility up to 200 000 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> (ref. 8), and a suspended monolayer MoS<sub>2</sub> crystal can have orders of magnitude enhancement in photoluminescence (PL) intensity over its on-substrate counterpart.<sup>9</sup> Moreover, suspended structures based on atomic layers are essential to enable 2D nanoelectromechanical systems (NEMS) such as ultrasensitive transducers and radio-frequency resonators. In particular, 2D materials possess remarkable mechanical properties such as high Young's modulus (*e.g.*,  $E_{\rm Y} \approx 1$  TPa for graphene<sup>10</sup>) and ultrahigh strain limits ( $\sigma_{\rm limit} \approx 30\%$  in black phosphorus<sup>11</sup>), making 2D materials highly promising for nanoscale sensors and actuators, and for integration with state-of-the-art NEMS to go across orders of magnitude length scales<sup>12</sup> for enabling new integrated nanosystems.

The recent discovery of giant magnetoresistance in WTe<sub>2</sub>,<sup>13</sup> resulting from the perfect balance between electron and hole populations,<sup>14</sup> has stimulated great interest in WTe<sub>2</sub> as a new layered material with important potential device applications. Similar to studies on other forerunners of 2D crystals, Raman spectroscopy has been a very important means for characterization. While earlier Raman studies of WTe<sub>2</sub> (on SiO<sub>2</sub> substrates) have identified up to 7 Raman modes, many theoretically predicted modes remain to be observed.<sup>15–19</sup> In addition, suspended WTe<sub>2</sub> structures can offer better control over material properties in WTe<sub>2</sub> such as mechanically engineering the magnetoresistance through manipulating the strain. Therefore, it is fundamentally important to systematically characterize the material properties including the mechanical properties of suspended WTe<sub>2</sub> crystals.

In this work, we fabricate both on-substrate and suspended WTe<sub>2</sub> structures and investigate both the lattice vibration *via* Raman spectroscopy and the coherent mechanical resonances of the entire suspended device structures. We observe a total of 12 Raman modes, all predicted by theoretical calculations,<sup>15–19</sup> and their evolution over number of layers. We also extract WTe<sub>2</sub> crystals' mechanical properties such as the Young's modulus ( $E_Y$ ) and pre-tension levels from nanomechanical resonance measurements.

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#### Raman characterization of single- and few-layer WTe<sub>2</sub>

Fig. 1a and b show the WTe<sub>2</sub> crystal structure. The atomic layers stack along the *c*-axis, and within each atomic layer the tungsten (W) atoms (lined up along the *a*-axis) are off-centered from the higher symmetry sites (Fig. 1b), forming the distorted octahedral structure:<sup>20</sup> the W atoms are sandwiched between two Te atomic sheets; the three nearest Te atoms from each sheet form a triangular pyramid with the W atom, with the two resulting opposing pyramids rotated 180° (along the *c*-axis) from each other.

The Raman measurement system used in this study is integrated into a home-built optical interferometer for ultrasensitive detection of the motions of the suspended samples, as illustrated in Fig. 1c (see Methods section). Some of the representative samples are displayed in Fig. 2. We have carefully prepared and verified multiple single-layer (1L) flakes (Fig. 2a and more samples similar to this), and examined their Raman signatures, which repeatedly show 3 peaks in the 70–400 cm<sup>-1</sup> range (see detailed zoom-in plots in Fig. 2b). The 1L flakes exhibit clear features and this facilitates their identification reliably, through optical image contrast (as compared to other thicknesses, see Fig. 2a and c for examples), Raman spectra (clearly different from data from 2L and other few-layer flakes), and AFM imaging. All the 1L flakes are meticulously fabricated and promptly transferred into a vacuum chamber for timely and repeated measurements (see Methods section). Fig. 2c-g demonstrate optical images and AFM results from typical few-layer samples.

Fig. 2h shows Raman spectra recorded from a six-layer (6L) WTe<sub>2</sub> flake with 12 Raman modes. To precisely determine the peaks' positions, we fit each peak with a Lorentzian curve, which gives Raman modes at 80.8, 86.9, 89.6, 110.4, 117.6, 120.8, 131.4, 134.8, 160.3, 163.9, 212.0, and 213.9 cm<sup>-1</sup> (referred to as P1–P12 hereafter), as well as the full width at half maximum (FWHM) values of these peaks in the range of ~1.7 to 2.8 cm<sup>-1</sup>. This is in clear contrast to previously reported Raman measurements in air, where there are only no more than 7 Raman modes with much larger FWHM values (up to 10 cm<sup>-1</sup>).<sup>15–19</sup> These results confirm that a comparatively high crystal quality of our WTe<sub>2</sub> flakes is preserved throughout the measurements.

We compare our measured Raman results with theoretical predictions,<sup>15–19</sup> in which A<sub>1</sub>, A<sub>2</sub>, B<sub>1</sub>, and B<sub>2</sub> Raman modes are expected for WTe<sub>2</sub>. In our measurement, it is expected that only A<sub>1</sub> and A<sub>2</sub> Raman modes would appear since the laser polarization is mostly orthogonal to the crystalline *c*-axis. In particular, Raman modes at P1 (80.8 cm<sup>-1</sup>), P10 (163.9 cm<sup>-1</sup>),



Fig. 1 Crystal structure of  $WTe_2$  and the measurement system. (a) Side view and (b) top view of the  $WTe_2$  crystal structure (distorted octahedral). Red and blue spheres represent W and Te atoms respectively. (c) Combined Raman spectroscopy/interferometry measurement system. LPF, BPF, PD, and BS represent a long-pass filter, a band-pass filter, a photodetector and a beam splitter, respectively. All measurements are performed with samples being in vacuum.



**Fig. 2** Raman spectroscopy results of single-layer (1L) and few-layer WTe<sub>2</sub>. (a) Optical image of representative 1L and few-layer WTe<sub>2</sub> flakes on the 290 nm SiO<sub>2</sub>/Si substrate. Yellow numbers label the Raman measurement positions on 1L flakes. Scale bar: 5  $\mu$ m. (b) Measured Raman signal from 1L WTe<sub>2</sub> flakes. (c) Optical image of exfoliated WTe<sub>2</sub> flakes (the numbers of layers are labeled) on the 290 nm SiO<sub>2</sub>/Si substrate. Scale bar: 5  $\mu$ m. (d and e) AFM images of areas outlined with (d) a blue box and (e) a red box in (c). Scale bars: 1  $\mu$ m. (f and g) AFM traces along dashed lines in (d) and (e), respectively. (h and i) Measured Raman spectra from (h) 6L and (i) 2L WTe<sub>2</sub>, with a Lorentzian fit. All peaks are labeled. (j) Evolution of Raman spectra from 8L to 1L. The data from 207 cm<sup>-1</sup> to 222 cm<sup>-1</sup> is zoomed in (k) to clearly show the evolution of P11 and P12 Raman modes. (i–o) Thickness dependence of Raman shift for all 12 modes.

P11 (212.0 cm<sup>-1</sup>), and P12 (213.9 cm<sup>-1</sup>) exhibit high intensity among measured peaks and are well matched to the predicted Raman modes.<sup>15–18</sup> For some of the  $B_1$  and  $B_2$  Raman modes that would not be favorably excited in our measurement scheme if considered only theoretically, we are still able to measure them due to out-of-plane polarization induced by the high numerical aperture (NA) of the microscope objective<sup>21</sup> in our experimental configuration.

By measuring crystals of different thicknesses, we explore the thickness dependence of Raman modes. Fig. 2i shows that the Raman spectrum of 2L WTe<sub>2</sub> differs from that of multilayer flakes. We observe 6 clear peaks: P1 (86.1 cm<sup>-1</sup>), P4 (109.0 cm<sup>-1</sup>), P5 (120.1 cm<sup>-1</sup>), P8 (136.1 cm<sup>-1</sup>), P10 (164.0 cm<sup>-1</sup>) and P12 (215.9 cm<sup>-1</sup>). In addition, FWHM values range from 3.5 to 10 cm<sup>-1</sup>, showing broadening compared with thicker samples. In the 1L WTe<sub>2</sub> flakes (Fig. 2a), the 3 Raman peaks exhibit larger FWHM values (7–22 cm<sup>-1</sup>) (see Fig. 2b, j and k) than in thicker layers. Fig. 2j shows the evolution of Raman spectra from 8L down to 1L WTe<sub>2</sub>. We observe that the intensities of P1 to P8 remain mostly unchanged from 8L down to 4L, but significantly decrease in 3L to 1L WTe<sub>2</sub>. This thickness dependence of Raman intensities in P1 to P8

(remain mostly unchanged from 8L down to 4L, and then quickly decrease down in 3L and thinner WTe<sub>2</sub>) may be explained by space group evolution from bulk ( $C_{2v}$ ) to 1L ( $C_{2h}$ ) WTe<sub>2</sub>. Theoretical calculations predict that the space group transition allows only a few Raman modes in 1L WTe<sub>2</sub>;<sup>16,18</sup> this may have contributed to significantly reducing the intensities of P1 to P8 modes when the thickness is reduced down to 3L, 2L, and 1L. Similar space group evolution is also theoretically predicted in other TMDCs (*e.g.*, WSe<sub>2</sub>, MOSe<sub>2</sub>, WS<sub>2</sub>, and MOS<sub>2</sub>) from bulk to few-layer and monolayer structures.<sup>22</sup>

Another important finding is the intensity evolution of P11 and P12 modes. Interestingly, the Raman peak around ~215.9 cm<sup>-1</sup> is composed of two peaks (P11 and P12) with unique thickness dependence of Raman intensity (Fig. 2k). In this pair, P11 is very strong in 8L, and then gradually declines from 8L to 4L, and is eventually too small to measure in 3L to 1L. This is similar to the behavior of P8. Meanwhile, P12 intensity increases as the thickness decreases, and becomes dominant in 1L to 3L WTe<sub>2</sub>. We attribute these observations to the transition of the space group in WTe<sub>2</sub>.<sup>16,18</sup>

In addition, we observe a clear and distinctive thickness dependence in the Raman shift for all 12 peaks (Fig. 2l–o). The thickness dependence of the 12 modes exhibits three trends: softening (P4, P7, and P9), stiffening (P1, P5, P8, P11 and P12) and invariant (P2, P3, P6, and P10) as the WTe<sub>2</sub> thickness decreases from 8L to 1L. Among all these Raman modes, we find that P1 has the greatest shift ( $\Delta \omega \approx 7 \text{ cm}^{-1}$ ), consistent with theoretical calculations.<sup>16</sup> This, together with the relative intensity and frequency changes in P11 and P12, provides clear finger-prints for determination of number of layers in WTe<sub>2</sub> samples.

We further compare the Raman shift over sample thickness with theoretical predictions. 2H-MoS<sub>2</sub>, the best-studied hexagonal TMDC, has two major Raman modes,  $E_{2g}^{1}$  (in-plane) and  $A_{g}^{1}$  (out-of-plane).<sup>23</sup>  $E_{2g}^{1}$  exhibits stiffening and  $A_{g}^{1}$  shows softening as thickness decreases. Unlike MoS<sub>2</sub>, theory predicts that Raman modes in WTe<sub>2</sub> are not confined to individual directions:<sup>16,19</sup> in particular, modes P6, P8 and P12 consist of both in-plane and out-of-plane motions, thus showing both stiffening and softening, as we have observed. Theory also predicts that mode P10 has displacement purely along the W atomic 1D chains, and is insensitive to crystal thickness,<sup>16,19</sup> which is also consistent with our observations.

We now examine the Raman spectra from both suspended and on-substrate regions of the same WTe<sub>2</sub> flakes. Fig. 3a shows the Raman spectra from a 13L WTe<sub>2</sub> crystal. In this sample, the WTe<sub>2</sub> flake partially covers the microtrench, making a leaking aperture. Hence, when we locate it in the vacuum chamber, the pressure levels both inside and outside the cavity quickly equilibrate to vacuum. We find a slight FWHM narrowing in suspended WTe<sub>2</sub> Raman modes compared to those from on-substrate WTe<sub>2</sub>. This may be attributed to reduced coupling to the substrate and minimal damping from air molecules. In contrast, a completely sealed 10L WTe<sub>2</sub> diaphragm (see Fig. 3b), which sustains atmospheric pressure inside the cavity when placed in the vacuum chamber, exhibits a slightly broader FWHM in all the Raman modes from sus-



Fig. 3 Measured Raman spectra of both suspended and on-substrate WTe<sub>2</sub> from (a) 13-layer (13L) and (b) 10-layer (10L) samples. Insets show the optical microscopy images, with the colored box showing the location where data (plotted in corresponding colors) are taken. Fitting is shown for data in (a) and (b). Scale bars: 2  $\mu$ m.

pended WTe<sub>2</sub> than those measured from on-substrate regions on the same flake, suggesting that damping from air molecules may play a role in Raman scattering of suspended WTe<sub>2</sub>.

#### Properties of suspended WTe2 via resonant measurements

We investigate resonance characteristics of suspended WTe<sub>2</sub> structures. Fig. 4a–d show the measured resonances from WTe<sub>2</sub> circular drumhead resonators of varying dimensions, with resonance frequencies of 8.6–32.1 MHz, all in the high frequency (HF) and very high frequency (VHF) bands, and quality (Q) factors of 67–249. We summarize resonance frequencies and Q values of all the measured devices in Fig. 4e and f.

We further perform analytical modeling on frequency scaling and compare the results with the measurement results. The fundamental-mode resonance frequency  $f_0$  of the WTe<sub>2</sub> resonators can be expressed as:<sup>24,25</sup>

$$f_0 = \left(\frac{kd}{4\pi}\right) \sqrt{\frac{16D}{\rho_{\rm 2D}d^4}} \left[ \left(\frac{kd}{2}\right)^2 + \frac{\gamma d^2}{2D} \right]. \tag{1}$$

Here,  $\rho_{2D}$  is the area density ( $\rho_{2D} = \rho_{3D} \times t$ , where  $\rho_{3D} =$  9430 kg m<sup>-3</sup> for WTe<sub>2</sub>), *k* a modal parameter (calculated numerically),  $\gamma$  the built-in tension or pre-tension, and  $D = E_{\rm Y} t^3 / [12(1 - \nu^2)]$  the flexural rigidity ( $E_{\rm Y}$ : Young's modulus;



Fig. 4 Resonance characteristics of suspended WTe<sub>2</sub> devices. (a–d) Measured fundamental resonances from devices with dimensions of (a)  $d \approx 5 \mu m$ ,  $t \approx 27 nm$ , (b)  $d \approx 5 \mu m$ ,  $t \approx 17 nm$ , (c)  $d \approx 3 \mu m$ ,  $t \approx 13 nm$ , and (d)  $d \approx 1.7 \mu m$ ,  $t \approx 8 nm$ , respectively. Dashed lines show fits to the damped harmonic resonator (from which Q values are extracted). Insets show optical microscopy images of each device. All scale bars: 2  $\mu m$ . (e) Resonance frequencies and (f) quality factors vs. device dimensions are summarized for all the measured devices (color symbol: black:  $d < 2 \mu m$ ; red:  $d > 2 \mu m$ ; blue: partially covered device).

 $\nu$ : Poisson's ratio). Since there is no experimental data for the exact Poisson's ratio of WTe<sub>2</sub>, we have used the value of  $\nu = 0.16$  predicted by theoretical calculations.<sup>26</sup> In the  $\frac{\gamma d^2}{D} \rightarrow \infty$  tension-dominant limit (or  $\frac{\gamma d^2}{D} \rightarrow 0$  modulus-dominant limit), eqn (1) approaches a membrane (or disk) model (see dashed lines in Fig. 5).

We extract Young's modulus and built-in tension levels of the WTe<sub>2</sub> resonators by comparing measurement results with analytical calculations (eqn (1)). Fig. 5 plots the frequency scaling of WTe<sub>2</sub> resonators using eqn (1) with diameters of 1.7  $\mu$ m, 3  $\mu$ m, and 5  $\mu$ m, clearly showing the elastic transition from the 'membrane limit' (tension dominanting) to the "disk limit" (flexural rigidity dominanting) as the crystal thickness increases. The resonantly tested devices (with thickness from 8 nm to 27 nm) operate in the transition regime, where their resonance frequencies are determined by both Young's modulus and the pre-tension. From the data we extract the averaged Young's modulus of  $E_{\rm Y} \approx 80$  GPa with a standard deviation of  $\sigma \approx 30$  GPa, and built-in pre-tension levels of ~0.05–0.5 N m<sup>-1</sup>. These results are consistent with theoretically predicted Young's modulus of WTe2.27,28 We note that in this mixed elasticity model, it is more important to find both the tension and  $E_{\rm Y}$  values. In order to determine  $E_{\rm Y}$  more precisely, we shall resort to devices that are completely in the disk regime, and are almost independent of tension. In our experi-



**Fig. 5** Frequency scaling of circular drumhead WTe<sub>2</sub> resonators. For each color, the upper solid line represents calculated resonance frequency with a tension level of  $\gamma = 0.5$  N m<sup>-1</sup> and the lower one represents  $\gamma = 0.05$  N m<sup>-1</sup>. The shadowed region indicates the tension levels in between. Dashed lines show membrane and plate limits of  $d \approx$ 5 µm resonators. Squares, triangles, and circles show measured data from devices of  $d \approx 1.7$  µm, 3 µm and 5 µm, respectively.

ment, we have not seen any thickness dependence of Young's modulus. Further, we have verified that the uncertainty of Poisson's ratio does not give a noticeable deviation to our frequency scaling and Young's modulus estimation. In fact, using different Poisson's ratio values (such as using  $\nu = 0.3$  to replace  $\nu = 0.16$ ) in eqn (1) only generates negligible differences.

# Conclusions

We have characterized both on-substrate and suspended fewlayer WTe<sub>2</sub> films by Raman spectroscopy. For the first time, we have observed a total of 12 Raman modes with small FWHM values in few-layer WTe2, with new modes and features that have not been reported in previous Raman measurements. All the 12 peaks exhibit distinctive thickness dependence (stiffening, softening, or invariant) as crystal thickness decreases from 8L to 1L, which can be used as an effective 'thickness indicator' for WTe2 flakes. These multimode Raman spectra can thus be collectively examined as characteristic fingerprints or signatures for single- and few- to multi-layer WTe<sub>2</sub>. We have also demonstrated the first HF and VHF WTe2 resonators, and by combining the measured resonance responses with frequency scaling analysis, we extract Young's modulus of these WTe<sub>2</sub> flakes ( $E_{\rm Y} \approx 80$  GPa) along with their pre-tension levels  $(\sim 0.05-0.5 \text{ N m}^{-1})$ . These results open up new opportunities for engineering nanomechanical WTe2 devices, such as strainengineered and resonantly tuned magnetoresistance sensors.

# Methods

### Single-crystal WTe<sub>2</sub> synthesis

Single crystal WTe<sub>2</sub> is grown by a chemical transport method.<sup>14</sup> Stoichiometric Te powder and W powder are put in a sealed tube filled with Br<sub>2</sub>. The tube is placed in a doublezone furnace with a temperature gradient of 100 °C between 750 °C and 650 °C. After one week, a large single crystal is synthesized. While other TMDCs, such as  $MoS_2$  and  $WSe_2$ , exist in the 2H crystal structure, WTe<sub>2</sub> exists in a distortion octahedral structure, also known as 1T' structure, under ambient conditions. The thickness of devices is confirmed by atomic force microscopy (AFM) with tapping mode. Fig. 2b–e show AFM images and height traces.

#### Suspended device fabrication

 $WTe_2$  flakes are exfoliated from the bulk  $WTe_2$  crystal onto 290 nm SiO<sub>2</sub> on the Si substrate. Suspended devices are fabricated using a dry-transfer method:<sup>29</sup>  $WTe_2$  flakes are first exfoliated onto PDMS stamps and then transferred onto prepatterned microtrenches. Since it is reported that ultrathin  $WTe_2$  could degrade quickly in air,<sup>16,19</sup> we store devices in a vacuum immediately after fabrication.

#### Raman scattering measurements

Raman measurements are performed using a customized micro-Raman system that is integrated into an optical inter-

ferometric resonance measurement system (Fig. 1c). WTe<sub>2</sub> flakes are preserved in a vacuum chamber with an optical window. The 532 nm laser is focused on the WTe<sub>2</sub> flakes in the vacuum chamber using a 50× microscope objective. The typical laser spot size is  $\sim 1 \ \mu m$  and the laser power is limited to below 200 µW to avoid excessive laser heating. Raman scattered light from the sample is collected in backscattering geometry and then guided to a spectrometer (Horiba iHR550) with a 2400 g mm<sup>-1</sup> grating. The Raman signal is recorded using a liquid-nitrogen-cooled CCD. The spectral resolution of our system is  $\sim 1 \text{ cm}^{-1}$ . Typical Raman spectrum measurement range for WTe<sub>2</sub> is 70 to 400  $\text{cm}^{-1}$ . Unlike measurements that are conducted in air, we measure Raman scattering of WTe<sub>2</sub> in a vacuum ( $p \sim 10$  mTorr). This vacuum environment can reduce effects from surface adsorbents such as O<sub>2</sub> and H<sub>2</sub>O which may lead to WTe<sub>2</sub> crystal degradation.

#### Interferometric resonance measurements

We study WTe<sub>2</sub> nanomechanical resonances using an ultrasensitive laser interferometry system (Fig. 1c). We photothermally excite resonant motions in suspended WTe<sub>2</sub> structures using an amplitude modulated 405 nm blue laser. To avoid excessive heating, the blue laser is focused ~5  $\mu$ m away from the devices and power is limited to below 300  $\mu$ W. The modulation depth and frequency of the 405 nm laser are controlled by a network analyzer (HP3577A), sweeping from 1 MHz to 50 MHz. A 633 nm red laser with an average power of 600  $\mu$ W is focused onto the center of the WTe<sub>2</sub> devices to detect the resonant motion. Typical laser spot sizes are ~5  $\mu$ m and ~1  $\mu$ m for the 405 nm and 633 nm lasers, respectively. The output signal in the frequency domain is recorded by the same network analyzer.

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