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Epitaxial growth of monolayer PdTe$_2$ and patterned PtTe$_2$ by direct tellurization of Pd and Pt surfaces

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Abstract

Two-dimensional (2D) palladium ditelluride (PdTe$_2$) and platinum ditelluride (PtTe$_2$) are two Dirac semimetals, which have fascinating quantum properties such as superconductivity, magnetism and topological order and show the promising applications in future nanoelectronics and optoelectronics. However, the synthesis of PdTe$_2$ and PtTe$_2$ monolayers (MLs) is hindered by a strong interlayer coupling and orbital hybridization. In this study, we demonstrate an efficient synthesis of PdTe$_2$ and PtTe$_2$ MLs. Large-area and high-quality MLs of PdTe$_2$ and patterned PtTe$_2$ were epitaxially grown on the Pd(111) and Pt(111) surfaces by direct tellurization in ultra-high vacuum. This was confirmed by x-ray photoelectron spectroscopy, low energy electron diffraction and scanning tunnelling microscopy. PdTe$_2$ ML demonstrated high thermal stability showing no decomposition sign after annealing at 470 °C. A well-ordered (2 × 2) PtTe$_2$ structure with Kagome lattice was observed on Te/Pt(111) surface following annealing at 200 °C in UHV, where the (2 × 2) pattern was formed by Te atom vacancies. PtTe$_2$ multilayer film was prepared as well, and it demonstrated the excitation of the Dirac plasmons as measured by high-resolution electron energy loss spectroscopy. The direct tellurization offers the simple and reliable protocol for the preparation of the ML of PdTe$_2$ and patterned PtTe$_2$, and this opens new opportunities for quantum phenomena research and for practical optoelectronics applications.

1. Introduction

Two-dimensional (2D) noble transition metal dichalcogenides (NTMDs) are a subgroup of the 2D transition metal dichalcogenides (TMDCs), which exhibit the drastically different properties from most TMDCs [1]. NTMDs demonstrate a strong thickness-dependence of the band structure, high mobility, in-plane anisotropy and high air stability; these offer the exciting opportunities in nanoelectronics, optoelectronics and catalysis [2–9]. Type II Dirac fermions with superconductive properties were shown in PdTe$_2$, the coexistence of the superconductivity and the topological states at the 2D level creates a possibility to observe Majorana fermions [10–13]. The excitation of the 3D Dirac plasmons was observed in PtTe$_2$ and this makes this material a promising candidate for optoelectronic applications [14–16]. Both PdTe$_2$ and PtTe$_2$ show thickness-dependence of the band structure [17], which gives an opportunity to tune the electrical and optical properties of the materials. It is highly desirable to scale down the high-quality PdTe$_2$ and PtTe$_2$ to a monolayer (ML).

Unlike other TMDCs, PdTe$_2$ and PtTe$_2$ show a strong interlayer interaction due to their specific electronic configurations, where the d levels are nearly full occupation and the p levels are highly hybridized [17, 18]. This makes challenging to obtain ultrathin
2D layers using the ‘top-down’ methods such as a mechanical exfoliation. The traditional methods such as molecular beam epitaxy (MBE), chemical vapor deposition (CVD) and pulsed laser deposition were successfully used to grow different 2D materials demonstrating the promising ‘bottom-up’ approach [19–21]. Recently, the PdTe2 and PtTe2 flakes were synthesized by MBE, CVD and laser-assisted synthesis [22–25]. However, as prepared 2D flakes have the size in micrometre scale and present multilayer materials. Therefore, the synthesis of large-area, high-quality ML 2D films attracts significant attention. Large-area and high-quality ML of PtSe2 was realized on the Pt(111) by selenium evaporation and sequential annealing in ultra-high vacuum (UHV) [26, 27]. A (3 × 3) superstructure was detected pointing to a strong interaction between PtSe2 ML and the substrate [27]. Also, the intrinsically patterned 17/1H PtSe2 ML was observed during the epitaxial growth by controlling the concentration of defects [26].

Here we report in the first time the synthesis of the large-area PdTe2 and PtTe2 ML films by direct telluridization of the Pd(111) and Pt(111) surfaces. Using a one-step approach, we obtained high-quality ML films as confirmed by scanning tunnelling microscopy (STM), x-ray photoelectron spectroscopy (XPS) and low-energy electron diffraction (LEED). In the case of PtTe2, a (2 × 2) structure with Kagome lattice was observed demonstrating the well-ordered vacancies of Te atoms in the topmost layer of PtTe2. Multilayer of PtTe2 was also obtained by this method and the excitation of Dirac plasmons was detected by high-resolution electron energy loss spectroscopy (HREELS). The reliable synthesis of the PdTe2 and patterned PtTe2 MLs open new application opportunities in electronics and optoelectronics.

2. Method

All experiments were performed using an Omicron Surface Analysis Cluster, which was described elsewhere [28–30]. The setup consists of an UHV preparation chamber and UHV μ-metal analysis chamber with base pressures of 1 × 10−9 mbar and 5 × 10−11 mbar, respectively. Pd(111) and Pt(111) single crystals with 9 mm of diameter, 1 mm of thickness and with orientation accuracy <0.5° (MaTeCK GmbH) were cleaned by repeated cycles of Ar+ sputtering and annealing in UHV at 780 °C. The surface cleanliness was monitored by XPS, LEED, and STM. For each deposition experiment, the single crystals were freshly cleaned. Tellurium (99.999%, Sigma-Aldrich) was thermally evaporated using a home-built evaporator in the preparation chamber. The tellurium temperature in the evaporation regime was in the range of 290 °C–300 °C and the evaporation rate was controlled by the heater power. The substrate was kept at room temperature (RT) during Te deposition. The amount of deposited tellurium was verified by XPS. The details of thickness of deposited Te and deposition time are shown in figure S1 (available online at stacks.iop.org/2DM/8/045033/mmmedia). The sample temperature was measured by a K-type thermocouple attached to the manipulator part with which a sample holder was in a good thermal contact. To complete the telluride synthesis, a single crystal was annealed at the specified temperature for 10 min in UHV.

STM images (Omicron ambient temperature UHV STM) were collected at RT using electrochemically etched W and Pt-Ir tips at constant current (topographic) mode. In all experiments reported here, the tip was electrically grounded, meaning that at positive bias the electrons flowed from the tip to the sample. The STM images were analyzed using WSxM software [31].

XPS was acquired using a non-monochromatic Mg Kα x-ray radiation (hv = 1253.6 eV) at 150 W. High resolution spectra were recorded at constant pass energy of 20 eV using the electron energy analyzer–Omicron EAC 125 and the analyzer controller–Omicron EAC 2000. The resolution of the instrument/sample system, which was measured as the full width at half maximum of Pt 4f7/2 peaks of the clean Pt(111) crystal, was approximately 1.2 eV. Photoelectrons were collected at a 45° angle with respect to the surface normal. The parameters used for the calculation of practical electron attenuation lengths (EALs) using NIST database tool [32] are shown in table S1 in the supporting information (SI).

LEED measurements were conducted in the analyst chamber straight after sample preparation with a four-grid optics (Omicron LEED).

3. Results and discussion

PdTe2 and PtTe2 MLs were grown by deposition of Te on the Pd(111) and Pt(111) surfaces followed by annealing under UHV conditions. The schematic cartoon of the process is shown in figure 1. HREELS spectra confirm the formation of PdTe2 and PtTe2 (figure S2). On PdTe2, HREELS losses were observed at 15 meV and 25 meV in agreement with the optical modes reported for PdTe2 bulk crystal [33]. The mode of PtTe2 shows a single vibration at 21 meV, which also follows the phonon dispersion features of PtTe2 [34].

The different annealing temperature is required for PdTe2 and PtTe2 to get a flat and high-quality surface. Thus, a large-area PdTe2 ML film was obtained after annealing at 470 °C (figure 2(b)), whereas the reasonably flat surface of PtTe2 was obtained following annealing at 200 °C (figure 2(f)). For PdTe2, the surface annealed at low temperatures was covered...
by small irregularly shaped islands although STM showed the atomic arrangement corresponding to PdTe$_2$ (figure S3(a)). Below we focus on the conditions giving the best growth results, which corresponds to the annealing temperature of 470 °C. LEED of PdTe$_2$ ML obtained following 470 °C annealing shows a hexagonal diffraction pattern confirming a high crystallinity the (1 × 1) PdTe$_2$ structure over a large area (insets of figures 2(b) and S4(a)). Moreover, LEED analysis performed at the multiple spots is fully consistent with this conclusion (figure S4). LEED indicates that the PdTe$_2$ film has the same crystalline orientation across the entire surface. This allows to conclude that a large area of PdTe$_2$ ML of several mm size was prepared. The high-resolution STM image in figure 2(c) demonstrates the atomic structures of PdTe$_2$ with a hexagonal arrangement of the Te atoms in the topmost layer. An interatomic distance of 4.7 Å (figure 2(d)) corresponds to $\sqrt{3} \times \sqrt{3}$ arrangement for Pd(111) ($\sqrt{3} \times 2.75 = 4.76$ Å). The PdTe$_2$ lattice is rotated by 30° with respect to the underlying Pd(111) substrate, aligning one of the unit vectors of PdTe$_2$ with the $\langle 112 \rangle$ direction of the Pd(111) substrate (figure S5). The $\sqrt{3} \times \sqrt{3}$ arrangement could be thermodynamically driven to match the fcc or hcp sites of underlying Pd(111) substrate.

In the case of PdTe$_2$, a ML film was obtained following annealing at the temperature of 200 °C. The prepared surface was flat, only a few nanoparticles and the decoration of a monoatomic step was observed by STM (figure 2(f)). Unlike the (1 × 1) structure observed on PdTe$_2$/Pd(111), LEED displays a hexagonal diffraction (2 × 2) pattern on the PdTe$_2$ surface (inset of figure 2(f)). The STM image in figure 2(g) shows atomic vacancies, which look like dark spots, forming the hexagonal structure with a periodicity of 8.2 Å (the line profile in figure 2(h)), which is approximately two times by the expected lattice parameter for PdTe$_2$ [35, 36]. This (2 × 2) PdTe$_2$ represents the Kagome lattice as shown by two green triangles in figure 2(g). These atomic vacancies are the ordered defects due to missing Te atoms in the topmost layer of PdTe$_2$. The size of the (2 × 2) PdTe$_2$ structure approximately matches three times of Pt(111) lattice ($2 \times 4.1$ Å $\approx 3 \times 2.77$ Å = 8.31 Å). The (2 × 2) PdTe$_2$ is aligned with the underlying Pt(111) having one of the unit cell vectors parallel with the $\langle 110 \rangle$ direction of Pt(111). This was concluded based on the LEED observation (figure S6). Therefore, we can suppose that the generated Te vacancies rearrange into the (2 × 2) pattern, which is driven thermodynamically to match the fcc or hcp sites of the underlying Pt(111) lattice. According to STM, in the (2 × 2) structure, one of four Te atoms of the topmost layer is missing, therefore, the formal stoichiometry should be PtTe$_{1.75}$. The (2 × 2) structure demonstrates high thermal stability. LEED does not change and remains the (2 × 2) patterns following annealing at 500 °C in UHV (figure S7(a)). The atomic resolution STM image shows that the (2 × 2) PdTe$_2$ is still mainly intact although a few defects are generated (figures S7(b)–(d)). We would like to underline that the (2 × 2) structure is unique and only observed for PdTe$_2$ and not for PdTe. To the best of our knowledge, the (2 × 2) structure was not reported before.

Chemical sensitivity of XPS was used to confirm the formation of PdTe$_2$ and PtTe$_2$. Following Te deposition at RT on Pd(111), the centroids of the Pd 3d$_{5/2}$ and Pd 3d$_{3/2}$ peaks were at 335.6 eV and 340.9 eV, respectively, which shift towards higher binding energies (BEs) by 0.5 eV with respect to those of clean Pd(111) (figures 3(a) and S8(a)). This indicates the formation of the Pd–Te chemical bond right after Te deposition, and it points to a low activation barrier for the reaction between Pd and Te. The surface cleanliness is an important factor to promote the reaction. Impinging tellurium atoms can easily interact with the surface of the noble metal creating the chemical bond. The formation of PdTe$_2$ particles with the size of approximately 2 nm was detected following deposition as shown in figure S9. The annealing at 470 °C in UHV leads to the decreasing of the Te amount and, the centroid of the Pd 3d$_{5/2}$ peak shifts back to 335.1 eV. This is because the contribution of Pd(111) bulk dominates in the XPS signal and the contribution of PdTe$_2$ ML is difficult to be separated. On the other hand, the annealing results in the shift of the Te 3d peaks towards lower BEs by 0.2 eV, which points to the further transfer of the electron density from Pd to Te confirming the formation of PdTe$_2$ (figure 3(b)). In the case of Te/Pt(111), the annealing at 200 °C does not lead to the shift of the Pt 4f and Te 3d peaks (figures 3(c), (d) and S8(b)). For both PdTe$_2$ and PtTe$_2$, the Te 3d$_{5/2}$ peak at 573.3 eV (figures 3(a) and (c)) is higher by approximately 0.5 eV comparing to the bulk materials [23, 24]. This could be attributed to charge transfer between the epitaxial grown MLs and the substrates due to differences in the work functions [37, 38].

The thickness of the as-grown films was calculated using XPS data analysed with a XPS Thickness Solver tool from Nanohub [39]. XPS thickness model is discussed in detail elsewhere [30, 40]. This
approach was proposed by Fadley [41]. The equation for the overlayer thickness, \( t \), can be written as

\[
\frac{N_t(\theta)}{N_s(\theta)} = \frac{\rho_t \times \frac{d\Omega}{d\Omega_s} \times \Lambda_t(E_s) \times \cos \theta}{\rho_s \times \frac{d\Omega}{d\Omega_s} \times \Lambda_s(E_s) \times \cos \theta} \times \frac{1 - \exp \left( -\frac{1}{\Lambda_t(E_s) \times \cos \theta} \right)}{\exp \left( -\frac{1}{\Lambda_s(E_s) \times \cos \theta} \right)}
\]  

(1)

where \( N_t(\theta) \) and \( N_s(\theta) \) are the photoemission peak areas of the overlayer (\( s \) is for Te 3d in our case) and the substrate (\( s \) is for Pd 3d or Pt 4f in our case) at the given photoemission angle, \( \theta \), measured in respect to the surface normal. \( \rho_t \) and \( \rho_s \) are the numbers of atoms per unit volume (density) for the overlayer and substrate. \( \frac{d\Omega}{d\Omega_s} \) and \( \frac{d\sigma_s}{d\Omega} \) are differential cross-sections for the overlayer and substrate photoemission peaks. This numbers can be calculated using the Scofield cross sections [42] and the Reilman asymmetry parameter [43]. Instead of the inelastic mean free paths in the original [41], we use the EAL: \( \Lambda_t(E_t) \) for the photoelectrons originated in the overlayer and attenuated in the overlayer, \( \Lambda_t(E_s) \) for the photoelectrons originated in the substrate and attenuated in the substrate, and \( \Lambda_s(E_s) \) for the photoelectrons originated in the substrate and attenuated in the overlayer. All EALs were calculated using NIST SRD-82 [32]. Equation (1) was solved numerically using the Nanohub tool [39]. In the [40], the Nanohub tool was validated for the thickness calculation of TMDC materials such as MoS\(_2\), and the results were crosschecked by transition electron microscopy.

The intensity ratio between the photoemission peaks obtained from the MoS\(_2\) films such as Mo 3d, S 2s and S 2p and the Al 2p peaks from the substrate was used as the primary input parameter for the thickness calculation in the [40]. In our case, the photoemission peaks of Pt and Pd from NTMDs cannot be separated from substrates. Therefore, we have simplified the system. For the calculation, we have considered the PdT\(_2\) (PtTe\(_2\)) overlayer as Te overlayer as shown in figure S10. The as-calculated thicknesses of the Te overlayers were 2.37 Å and 2.48 Å for PdT\(_2\) and PtTe\(_2\), respectively. To verify if these numbers correspond to ML, the ML PtSe\(_2\) on Pt(111) was used as a reference because of the similar structural construction with our system. By following the recipe from [26, 27], ML PtSe\(_2\) was grown on Pt(111) confirmed by STEM, LEED and XPS (figure S11). Using the same calculation method, the thickness of Se overlay was 2.26 Å, which corresponds to the PtTe\(_2\) ML on Pt(111) and can be used as a reference value for our system. The ratio between 2.37 Å and 2.26 Å is 1.05, corresponding to 1.05 ML for PdT\(_2\) on Pd(111). The PtTe\(_2\) on Pt(111) estimated in the same way is 1.10 ML. Therefore, the conclusion is that in both cases the MLs of PdT\(_2\) and PtTe\(_2\) were successfully grown. On the other hand, no bilayer or multilayer areas were detected by STEM, also confirming the ML natures. The step height was approximately 2.2 Å (figure S12), matching a monoatomic step of Pd(111) or Pt(111) and
pointing to that PtTe₂ or PtTe₃ was grown epitaxially as a single layer.

The annealing temperature plays an important role in the PtTe₂ and PtTe₃ synthesis. Following the annealing at the temperature of 100 °C, the Te/Pd(111) and Te/Pt(111) surfaces are not flat and are covered with islands (figures 4(a) and S3(a)). The formation of the islands with an irregular shape indicates a non-thermodynamic equilibrium growth. Atomic structures of PtTe₂ can be resolved on top of the islands and on the terraces following the annealing even at of 100 °C (inset of figure S3(a) in the SI), further confirming the low activation barrier of the reaction between Pd and Te. Likely, the PtTe₂ ML film covers the surface as a ‘carpet’.

The Pt–Te interaction is more complex. Following the annealing at 100 °C, two atomic structures labelled with ‘I’ and ‘II’ were detected by STM (figure 4(a) and (b)). Structure I exhibits hexagonal atomic arrangements with the periodicity of 4.1 Å (figures 4(c) and (e)), which corresponds to the normal PtTe₂ lattice [35, 36], and we refer to this structure as a (1 × 1) PtTe₂. Typically, this phase is observed outside of the islands on the terraces. Structure II is the discussed above (2 × 2) PtTe₂ Kagome lattice (figure 4(d)). LEED patterns show bright (1 × 1) and weak (2 × 2) diffraction spots, confirming the coexistence of the two structures across a large area (inset of figure 4(b)). The (1 × 1) phase of PtTe₂ has higher surface density of Te comparing to the (2 × 2) structure. However, the latter is more thermodynamically stable; the (2 × 2) phase dominates completely following the annealing at 200 °C. Moreover, the 500 °C annealing in UHV did not affect much the (2 × 2) PtTe₂ phase, STM detects only a few atomic defects appeared on the surface (figure S7).

The PtTe₂ multilayer film was grown by the substantial increasing of the deposited amount of Te (figure 5(a)). The thickness calculated based on the XPS results was estimated to be approximately four PtTe₂ layers. The height of the step of multilayer PtTe₂ is 5.0 Å, (inset of figure 5(a)) corresponding to c lattice constant of PtTe₂ [14]. The PtTe₂ multilayer is characterized by the Pt 4f½ and 4f½ peaks at 71.6 eV and 74.9 eV, respectively (figure S13). These positions are shifted towards the higher BEs by 0.4 eV comparing to the ML case. Meanwhile, the Te 3d½ and 3d½ peaks are at 572.8 eV and 583.2 eV, which are shifted towards lower BEs by 0.5 eV comparing to the ML case. The BEs of the Te 3d peaks are consistent with the values reported in the literature for PtTe₂ [23]. This also confirms our hypothesis about the charge transfer between the Pt(111) substrate and the PtTe₂ ML. The atomic structure of the PtTe₂ multilayer has the periodicity of 4.1 Å (figure 5(b)) corresponding to the lattice constant of PtTe₂ [36]. LEED shows the typical (1 × 1) diffraction patterns (inset of figure 5(b)) indicating the high crystallinity across
Figure 5. Multilayer PtTe$_2$. (a) A STM topographic image of multilayer PtTe$_2$. The tunnelling conditions are $V_b = -1.1$ V, $I_t = 0.8$ nA. Inset: a height profile along the black dashed line in (a). (b) Atomic resolution STM image of the multilayer PtTe$_2$. The blue rhombus indicates a unit cell. The tunnelling conditions are $V_b = 0.9$ V, $I_t = 0.7$ nA. Inset: LEED patterns of the multilayer PtTe$_2$. The blue dashed circles indicate the $(1 \times 1)$ reflexes. (c) HREELS spectrum of the multilayer PtTe$_2$ (after background subtraction). The blue line is ‘guide for the eye’. The kinetic energy of the electron beam ($E_i$) is 5 eV. The incident angle ($\alpha_i$) and scattering angle ($\alpha_s$) for the HREELS measurement are 60$^\circ$ and 54$^\circ$, respectively.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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