Quantum Oscillations from Nontrivial States in Quasi-Two-Dimensional Dirac Semimetal ZrTe$_5$ Nanowires

Pei Yang$^1$, Wei Wang$^2$, Xiaoqian Zhang$^1$, Kejie Wang$^1$, Liang He$^1$, Wenqing Liu$^2$ & Yongbing Xu$^{1,2}$

Recently discovered Dirac semimetal ZrTe$_5$ bulk crystal, exhibits nontrivial conducting states in each individual layer, holding great potential for novel spintronic applications. Here, to reveal the transport properties of ZrTe$_5$, we fabricated ZrTe$_5$ nanowires (NWs) devices, with much larger surface-to-volume ratio than bulk materials. Quantum oscillations induced by the two-dimensional (2D) nontrivial conducting states have been observed from these NWs and a finite Berry phase of $\sim \pi$ is obtained by the analysis of Landau-level fan diagram. More importantly, the absence of the Aharonov-Bohm (A-B) oscillations, along with the Shubnikov-de Hass (SdH) oscillations, suggests that the electrons only conduct inside each layer. And the intralayer conducting is suppressed because of the weak connection between adjacent layers. Our results demonstrate that ZrTe$_5$ NWs can serve as a suitable quasi-2D Dirac semimetal with high mobility ($\sim 85000$ cm$^2$V$^{-1}$s$^{-1}$) and large nontrivial conductance contribution (up to 8.68%).

The simple binary compound ZrTe$_5$ is known as a thermoelectric material with layered structure. Recently it has been theoretically predicted as a 2D TI with a bulk bandgap of 0.4 eV$^1$ in the single layer form, while its three-dimensional (3D) version has been reported with many contradictory results such as 3D TI$^{2,3}$, 3D strong TI$^4$ and quasi-2D or 3D Dirac semimetal$^5$$^9$. Our previous study has suggested that the transport property of bulk ZrTe$_5$ is a collective behavior of many individual monolayers$^{10}$. However, the critical transport phenomena in NWs which have a much larger surface-to-volume ratio comparing to their bulk forms have not been fully studied.

Here, we have fabricated ZrTe$_5$ NWs devices to probe the electric transport from the nontrivial conducting states. We report the Shubnikov-de Hass (SdH) oscillations originated from the nontrivial states of each monolayer. The SdH interactions reveal a well-defined 2D Fermi surface lasting up to 20 K. The finite Berry phase of $\sim \pi$ with a high mobility of 85000 cm$^2$V$^{-1}$s$^{-1}$ clarifies the topological nontrivial nature. More importantly, the A-B oscillations were not observed when the magnetic field was applied along the current direction parallel to the NW. All of these suggest that the electrons only conduct within each layer and cannot hop between different layers, since the interlayer coupling between adjacent layers is too weak. According to the calculation by H. Weng et al., the binding energy of ZrTe$_5$ is only 12.5 meV, much lower than Bi$_2$Se$_3$ (27.6 meV) and very close to graphite (9.3 meV)$^9$.

**Results**

**Morphology and structural characterization of ZrTe$_5$ NWs.** Figure 1a exhibits the scanning electron microscope (SEM) image of a ZrTe$_5$ NW with a diameter of $\sim 290$ nm. The length of our ZrTe$_5$ NWs ranges from a few microns to tens of microns. Figure 1b is the tilted SEM of the NW with a tilt angle of 30°. It shows clearly the layer steps on the side walls.

---

1Jiangsu Provincial Key Laboratory for Nanotechnology, Collaborative Innovation Center of Advanced Microstructures, School of Electronic Science and Engineering, Nanjing University, Nanjing, 210093, P. R. China.
2York Nanjing Joint Centre for Spintronics and Nanotechnology, Departments of Electronics, The University of York, York, YO10 5DD, UK. Correspondence and requests for materials should be addressed to L.H. (email: heliang@nju.edu.cn) or Y.X. (email: yongbing.xu@york.ac.uk)
The crystallographic structure of ZrTe$_5$ is an orthorhombic layered structure$^{11}$ which is shown in Fig. 2a. Prism chains of ZrTe$_3$ (Te$^3$) is along the a-axis, and these prismatic chains are bonded via zigzag Te atoms (Te$^z$) along the c-axis to form a 2D sheet of ZrTe$_5$ in the a-c plane. The sheets of ZrTe$_5$ form a layered structure stacking along the b-axis. The primitive unit cell contains two formula units with two prismatic chains and two zigzag chains, as indicated by the dashed black square in Fig. 2a.

To perform the transmission electron microscopy (TEM)-energy dispersive X-ray spectrometry (EDX) analyses, the ZrTe$_5$ crystal NWs were dispersed in ethanol and deposited onto a carbon film supported by a 200 mesh, 3 mm diameter copper grid. Figure 2b reveals the atomic level details of the a-c plane of ZrTe$_5$. From the TEM image, we are able to determine the lattice constants of a and c are 0.4 nm and 1.4 nm, respectively. At the same time, from the X-ray diffraction (XRD) pattern (Fig. 2c), we can calculate the lattice constants $a = 0.398$ nm $b = 1.452$ nm and $c = 1.372$ nm, consistent with the result of TEM. The EDX spectrum detected Zr, Te, C and Cu, which were attributed to the NWs, the carbon film and copper grid (Fig. 2d). The Zr/Te atomic ratio in the EDX area was 0.28, which was much lower than that of the nominal composition. This discrepancy is probably due to the vacancies of Te atoms, similar to those reported in 3D TI, Bi$_2$Te$_3$ $^{12}$. The band dispersion obtained by ARPES measurement of bulk ZrTe$_5$ crystal$^8$ at 80 K is sketched in the inset of Fig. 2d. An almost linear E-K dispersion (as indicated by the yellow dashed lines) was observed near the $\Gamma$ point, suggesting the presence of Dirac fermions. Moreover, the energy dispersion shows a small gap opening rather than massless Dirac cone. Note that our ARPES result was done at 80 K, and the band diagram is going to shift down as the temperature decreases to lower values of 2–20 K$^{13}$, which results in the $E_F$ of our samples (S1 and S2) shifting up as indicated by the green lines at lower temperatures similar to that from the ref$^2$, as estimated from the quantum oscillations. The temperature dependent shift of $E_F$ is probably due to the change of lattice constants$^{1,14}$, as the temperature decreases.

**Electrical transport measurements of ZrTe$_5$ NW devices.** Figure 3a exhibits a four-probe device with Au contacts while the width and channel length are 3.2 $\mu$m and 4.6 $\mu$m, respectively. The temperature dependent longitudinal resistance $R_{xx}$ of S2 is shown in Fig. 3b. $R_{xx}$ demonstrates a peak at 125 K, known as the “resistivity anomaly”, close to the values reported in previous work ($\sim$60–170 K). This resistivity anomaly might be associated with a change in the electronic structure caused by thermal expansion, and the detailed physics is still under debating$^{8,13–30}$.

As we know, one of the persuasive proofs for characterizing nontrivial conducting states is regarded as SdH oscillations$^{8,29–32}$. According to this, low-temperature magneto transport measurements were carried out to demonstrate the nontrivial conducting states in ZrTe$_5$ NWs experimentally.

The angle dependent magneto transport properties of S1 are shown in Fig. 3c,d. Figure 3c exhibits the field dependent resistance for different $\theta$ ($0^\circ$, $30^\circ$, $45^\circ$, $60^\circ$ and $90^\circ$) from $-9$ T to $+9$ T at 2 K before subtracting the smooth background. Here $\theta$ is the tilt angle between the field direction B and the crystallographic b-axis, within b-a plane. The magnetoresistance, MR curves display pronounced SdH oscillations from 0° to 60°. While $MR(B) = (R(B) - R(0))/R(0)$, typically has a magnitude of 32% near 9 T, the amplitude of the SdH signal is small and amounts to only 0.4% of the total resistance. After subtracting a smooth background, $\Delta R_{xx}$ demonstrates much more clear oscillations. The inset of Fig. 3c shows the magnetic field B corresponding to the $\pi = 3$ minimum at varies rotation angles $\theta$, up to 60°. It follows $1/\cos(\theta)$ perfectly. This concludes that the quantum oscillations arise from a 2D Fermi surface.

**Quantum oscillations arising from the 2D nontrivial states.** The temperature dependent magneto transport properties of S2 after subtracting a smooth background is shown in Fig. 4a. The oscillatory part of $R_{xx}$ ($\Delta R_{xx}$) reveals periodic dependences with peaks (maxima) and valleys (minima) versus $1/B$, indicating there’s a well-defined Fermi surface$^{31,33,34}$. The magnetic field is perpendicular to both the c-axis and the charge current flow (a-axis) of the ZrTe$_5$ NW ($\theta = 0^\circ$). The SdH oscillations can be seen from 2 K up to 20 K. After fast Fourier transform (FFT) we can obtain a single oscillation frequency ($f_{SdH}$, 3.57 T). For a 2D system, the Onsager
formula: \( f_{\text{SdH}} = \left( \frac{h}{4 \pi^2 e} \right) S_F \), can describe the relation between SdH oscillation frequency and the cross section of the Fermi surface \( S_F \), where \( S_F = \pi k_F^2 \), \( k_F \) is the Fermi vector, \( e \) is the electron charge, and \( h \) is Planck constant.

The 2D surface carrier density \( n_{2D} \) can be calculated by \( n_{2D} = \frac{k_F^2}{4 \pi} \). Then we can extract \( k_F \) to be \( 0.0104 \) Å\(^{-1} \) by substituting \( S_F \) in \( f_{\text{SdH}} \), corresponding to \( n_{2D} = 0.86 \times 10^{11} \) cm\(^{-2} \).

The \( 1/B \) values of the maxima (hollow rectangles) and the minima (hollow circles) in \( \Delta R_{xx} \) versus Landau level index \( n \) are plotted in Fig. 4b. We extracted a finite intercept of \( 0.580 \pm 0.001 \), by linear fitting of the data, indicating a Berry phase of \( \sim \pi \), emphasizing the topological nature of the SdH oscillations. We have noticed that there’s a little discrepancy between the intercept and \( 1/2 \). The reason could be that for 3D or quasi-2D crystal, there is an additional phase shift determined by the dimensionality of the Fermi surface and the value changes from \( 0 \) for surface states (2D) to \( \pm \frac{1}{8} \) for bulk states (3D)\(^{36} \). Such inconsistence may also be attributed to the Zeeman splitting and/or the multiple Hall-channel contributions\(^{37} \).

The temperature-dependent amplitude of \( \Delta \sigma_{xx}(T) / \Delta \sigma_{xx}(0) \) can be written as \( \Delta \sigma_{xx}(T) / \Delta \sigma_{xx}(0) = \lambda(T) / \sinh(\lambda(T)) \), where \( \lambda(T) = 2 \pi^2 k_B T m_{\text{cycl}}/(\hbar e B) \), \( m_{\text{cycl}} \) is the cyclotron mass, \( h \) is the reduced Planck’s constant, and \( k_B \) is Boltzmann’s constant. After fitting the conductivity oscillation amplitude to the \( \Delta \sigma_{xx}(T) / \Delta \sigma_{xx}(0) \) equation, \( m_{\text{cycl}} \) is calculated to be \( 0.031 \) m\(_e \) (m\(_e \) is the free electron mass), which is shown in Fig. 4c. For a Dirac-like dispersion, \( E_F = \hbar k_V \) and also \( p_F = m_{\text{cycl}} V_F \), so \( m_{\text{cycl}} = E_F / V_F^2 \). This yields a Fermi level of \( \sim 26.64 \) meV above the Dirac point and a Fermi velocity of \( \sim 3.89 \times 10^5 \) m/s, which is in a good agreement with results reported by others\(^{39} \).

We can extract the transport lifetime of the surface states (\( \tau \)) by the Dingle plot\(^{30,31,33,40} \). Since \( \Delta R/R_0 \sim [\lambda(T)/\sinh(\lambda(T))] e^{-D} \), where \( D = 2 \pi^2 E_F / \tau e B V_F^2 \), the lifetime \( \tau \) can be obtained by the slope in Dingle plot by
The absence of A-B oscillations. Quantum interference effects, such as A-B oscillations\(^4\) associated with the surface states may occur for mesoscopic samples where the low-temperature mean free path is comparable to the sample dimensions. Theoretically, only half revolution around the perimeter of the NW (~390 nm, since...
the thickness \( t \) and width \( w \) of our NW are 100 nm and 290 nm respectively) is required for the interference effect of the A-B oscillations. Practically the mean free path \( l \) extracted from our results is \( \sim 583 \) nm, which is a lower estimation of the phase-coherent diffusion length in general. So, the phase-coherent diffusion length is long enough for the observation of A-B oscillations in the NW, if it exists.

Further calculation indicates that the cross-sectional area of the NW is \( S = w \times t = 2.9 \times 10^{-14} \) m\(^2\). Thus, the characteristic period of the A-B oscillations should be \( \Delta B = \Phi_0 / S = 0.143 \) T, where \( \Phi_0 = h/e \) is the flux quantum, \( S \) is the cross-sectional area of the NW, \( h \) is Planck's constant and \( e \) is the electron charge. Meanwhile, we can estimate the amplitude of the A-B oscillations, if exist. The unsuppressed amplitude of A-B oscillations should be in the order of \( e^2/h \) in conductance, which means when \( G_0 (= 1/R_0) \) changes in \( e^2/h \), the resistance after changing

**Figure 4.** Quantum oscillations arising from the 2D nontrivial states. (a) Temperature dependent SdH oscillations of ZrTe\(_5\) NWs at \( \theta = 0° \). The black solid lines mark the SdH valleys at Landau filling factors of 2, 3, 4 and 5, while the dash lines mark the peaks at 1.5, 2.5, 3.5 and 4.5. (b) Landau-level fan diagram. Linear fitting gives a nonzero intercept of 0.580, corresponding to a Berry phase of \( \sim \pi \). (c) Temperature dependence of the normalized conductivity amplitude \( \Delta \sigma_{xx}(T)/\Delta \sigma_{xx}(0) \). The solid red line is the best fit to \( \lambda(T)/\sinh(\lambda(T)) \). A magnetic field of 5.22 T was used to extract the cyclotron effective mass: \( \sim 0.031 m_e \). (d) Dingle plots of log \( [\Delta R/R_0]B\sinh(\lambda) \) versus \( 1/B \) at four different temperatures. Transport lifetime \( \tau \), mean free path \( l = V_F \tau \), and mobility \( \mu \) can be extracted from the best fit to log \( [\Delta R/R_0]B\sinh(\lambda) \).

**Table 1.** Estimated parameters from the SdH oscillations at \( T = 2 \) K.

| Sample | \( f_{	ext{eff}} \) (T) | \( n_{\text{SdH}} \) (10\(^{11}\) cm\(^{-2}\)) | \( m_{\text{SdH}} \) (m\(_e\)) | \( k_0 \) (Å\(^{-1}\)) | \( V_0 \) (10\(^{4}\) m\(^{-1}\)s\(^{-1}\)) | \( E_0 \) (meV) | \( \tau \) (10\(^{-12}\) s) | \( l \) (nm) | \( \mu \) (cm\(^2\) V\(^{-1}\)s\(^{-1}\)) |
|--------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| S1     | 8.58            | 2.08            | 0.037           | 0.0161          | 5.04            | 53.59           | 1.75            | 881             | 82915           |
| S2     | 3.57            | 0.86            | 0.031           | 0.0104          | 3.89            | 26.64           | 1.5             | 583             | 85083           |

**Table 2.** Estimated surface conduction percentage with zero magnetic field and at \( T = 2 \) K.

| Sample | \( G(\text{SdH}) \) (mS) | \( R(\text{total}) \) (Ω) | \( R_{\text{sheet}}(\text{total}) \) (Ω) | \( G_{\text{sheet}}(\text{total}) \) (mS) | \( G(\text{SdH})/G_{\text{sheet}}(\text{total}) \) |
|--------|-----------------|-----------------|-----------------|-----------------|-----------------|
| S1     | 2.76            | 453.7           | 9.07            | 11.02           | ~2.50%          |
| S2     | 1.18            | 460.9           | 73.74           | 13.56           | ~8.68%          |
should be $1/(1/R_0 + e^2/h)$, giving the $\Delta R = R_0 - R$. That is, $\Delta R = \Delta(1/G) \approx R_0 - 1/(1/R_0 + e^2/h) \approx 7.85 \Omega$, considering $R_0 \approx 454.1 \Omega$. However, for the magneto-conductance curves in the unit of $e^2/h$ at $\theta = 90^\circ (B/I)$, there's no A-B oscillations in our NWs (Fig. 3d).

After subtracted the smooth background (red lines in Fig. 3d), the magneto-conductance trace $\Delta G$ measured in a longitudinal field is shown in Fig. 3e. From this quantum magneto-conductance curve, there's no such oscillations consistent with the calculated A-B oscillations whose period should be 0.143 T. Meanwhile, the upper bound of $\Delta G$ is $-0.04 \ e^2/h$.

However, A-B oscillations are usually suppressed in cylindrical conductors, like our NWs. The main origins of this reduction are probably as follows. First, different slices of the metal cylinder (effectively 2D metal ring) generate A-B oscillations of random phases, canceling each other. Second, the electrons circle along the NW perimeter in a quasi-ballistic manner, but drift along the longitudinal direction of the NW in a diffusive manner (mean free path < the length of NW, $L_{NW}$). As probing the longitudinal conductance, A-B oscillation amplitude of conductance may be reduced due to the diffusive transport in longitudinal direction\cite{11}.

While, in 2014, Seung Sae Hong et al. from Prof. Yi Cui’s group have studied the effect of NW length on the A-B oscillations. Generally, $L_{NW}$ is comparable or longer than phase coherence length ($\lambda_p$). Especially at high temperature, $\lambda_p$ is expected to be much longer than $\lambda_p$. Therefore, if A-B oscillations are of random phase nature, oscillations of different segments ($L_{NM} \sim \lambda_p$) would be averaged out by additional factor ($L_{NM}/L_p(T)$)\cite{32, 13}. Then the amplitude of A-B oscillations in our experiments should be around 7.85 $\Omega^*$ (4.62 $\mu$m/583 nm) $\approx 2.79 \Omega$ near 454.1 T because of the suppression, corresponding to 0.3491 $e^2/h$, which is still much larger than our upper bound $(-0.04 \ e^2/h)$. Thus, to our detecting limit of $10^{-3} \ e^2/h$, there is no A-B oscillations.

The fast Fourier transform after background subtraction is also shown in Fig. 3f, which only has one pronounced peak at $0.337 \pm 0.0004 \ T^{-1}$, this number is not close to the A-B oscillation frequency of $6.993 \ T^{-1}$ ($\approx/0.143$ T) estimated from the cross-section area, which again confirms the absence of A-B oscillations. After carefully analyzing our data, we believe this oscillation could originate from the systematical errors of our system, probably because of the digital noises in our measurement system or from the universal conductance fluctuations\cite{14}. If we changed the scanning speed or data acquisition speed, the background oscillation frequencies scale with it.

The absence of the A-B oscillations may be attributed to no conducting channels at the sidewalls, because of the weak interlayer coupling, as shown in Fig. 1c. Thus, there is no path for the Dirac electrons to travel around the perimeter of the NW. Therefore, we have provided another piece of evidence that ZrTe$_5$ NW is a quasi-2D Dirac semimetal with very weak interlayer coupling, which is in a good agreement with W. Wang et al.’s conclusion\cite{15}.

**Discussion**

In summary, we have fabricated the ZrTe$_5$ NWs devices with four-terminal geometry and measured the magnetoresistance properties under varied temperatures and angles. The angle-dependent SdH oscillations have unambiguously shown nontrivial conducting states with high carrier mobility ($\approx 85000 \ cm^2/V^{-1}s^{-1}$), and they contribute up to 8.68% of the total conductance. Since the metallic properties under very low temperatures of our NWs and the non-zero Berry phase we obtained, we believe our ZrTe$_5$ NWs belong to the Dirac semimetal. In addition, the negative magnetoresistance properties observed by Qiang Li et al.\cite{16} confirm again that ZrTe$_5$ should be a Dirac semimetal. The absence of A-B oscillations suggests that there’s no path for the electrons to travel around the perimeter of our NWs. This together with the SdH oscillations suggest that there is only weak interlayer coupling between adjacent layers of the ZrTe$_5$ NWs.

**Methods**

The ZrTe$_5$ crystal was grown by chemical vapor transportation (CVT) method. ZrTe$_5$ was firstly exfoliated on scotch tape and then transferred onto 300 nm/300 nm SiO$_2$/p-Si substrate. Conventional photolithography was used to pattern the ZrTe$_5$ NWs into a micron-scale four-terminal device followed by a subsequent dry etching ($5–15$ s Ar ion etching). Four paralleled electrodes ($50$ nm Au) were defined by e-beam evaporation and the lift-off process. To study the 2D nontrivial conducting states of our ZrTe$_5$ NWs, magneto transport measurements were conducted. A schematic diagram of the device structure is shown in Fig. 3a. The current is along the a-axis as shown by the yellow arrows. In order to study the angle-dependent and the temperature-dependent magnetoresistance properties, we have fabricated two devices with same geometry called S1 and S2, respectively.

**References**

1. Weng, H. & Dai, X. & Fang, Z. Transition-Metal Pentatelluride ZrTe$_5$ and HfTe$_5$: A Paradigm for Large-Gap Quantum Spin Hall Insulators. Phys. Rev. X 4, 011002 (2014).
2. Wu, R. et al. Evidence for Topological Edge States in a Large Energy Gap near the Step Edges on the Surface of ZrTe$_5$. Phys. Rev. X 6, 021017 (2016).
3. Li, X. B. et al. Experimental Observation of Topological Edge States at the Surface Step Edge of the Topological Insulator ZrTe$_5$. Phys. Rev. Lett. 116, 176803 (2016).
4. Manzoni, G. et al. Evidence for a Strong Topological Insulator Phase in ZrTe$_5$. Phys. Rev. Lett. 117, 237601 (2016).
5. Li, Q. et al. Chiral magnetic effect in ZrTe$_5$. Nature Phys. 12, 550–554 (2016).
6. Zheng, G. et al. Transport evidence for the three-dimensional Dirac semimetal phase in ZrTe$_5$. Phys. Rev. B 93, 115414 (2016).
7. Chen, R. Y. et al. Magneto-infrared Spectroscopy of Landau Levels and Zeeman Splitting of Three-Dimensional Massless Dirac Fermions in ZrTe$_5$. Phys. Rev. Lett. 115, 176404 (2013).
8. Chen, R. Y. et al. Optical transport study of three-dimensional Dirac semimetal ZrTe$_5$. Phys. Rev. B 92, 075107 (2015).
9. Yuan, X. Y. et al. Observation of quasi-two-dimensional Dirac fermions in ZrTe$_5$, NPG Asia Mater. 8, e325 (2016).
10. Wang, W. et al. Evidence for Layered Quantized Transport in Dirac Semimetal ZrTe$_5$. Sci. Rep. 8, 5125 (2018).
11. Fjellvåg, H. & Røjkons, A. Structural properties of ZrTe$_5$ and HfTe$_5$ as seen by powder diffraction. Solid State Commun. 60, 91–93 (1986).
12. Messenger, A., Sayah, D., Tedesco, J. C. & Boyer, A. Crystal growth of Bi$_2$Te$_3$ on single-crystal substrate Nb$_2$Te$_5$, by molecular beam epitaxy. Int. J. Electron. 77, 291–300 (1994).
13. Zhang, Y. et al. Electronic evidence of temperature-induced Lifshitz transition and topological nature in ZrTe$_5$. Nature Commun 8, 15512 (2017).
14. Xiong, H. et al. Three-dimensional nature of the band structure of ZrTe5, measured by high-momentum-resolution photoemission spectroscopy. Phys. Rev. B 95, 195119 (2017).
15. Stanescu, T. D., Sau, J. D., Lutchyn, R. M. & Sarma, S. D. Proximity effect at the superconductor–topological insulator interface. Phys. Rev. B 81, 241310 (2010).
16. Wetting, T. J. et al. Giant anomalies in the resistivities of quasi-one-dimensional ZrTe5 and HfTe5. Bull. Am. Phys. Soc. 25, 340 (1980).
17. Okada, S. et al. Giant resistivity anomaly in ZrTe5. J. Phys. Soc. Jpn 49, 839 (1980).
18. Jones, T. E. et al. Thermoelectric power of HfTe5 and ZrTe5. Solid State Commun. 42, 793–798 (1982).
19. McIlroy, D. N. et al. Observation of a semimetal–semiconductor phase transition in the intermetallic ZrTe5. J. Phys.: Condens. Matter 16, 359–365 (2004).
20. DiSalvo, F. J. et al. Possible phase transition in the quasi-one-dimensional materials ZrTe5 and HfTe5. Phys. Rev. B 24, 2935 (1981).
21. Okada, S. et al. Negative evidences for charge/spin density wave in ZrTe5. J. Phys. Soc. Jpn 51, 460 (1982).
22. Rubin, M. ZrTe5 and HfTe5: possible polaronic conductors. Phys. Rev. B 60, 1627 (1999).
23. Manzoni, G. et al. Ultrafast optical control of the electronic properties of ZrTe5. Phys. Rev. Lett. 115, 207402 (2015).
24. Zhou, Y. H. et al. Pressure-induced semimetal to superconductor transition in a three-dimensional topological material ZrTe5. Proc. Natl Acad. Sci. USA 113, 2904–2909 (2016).
25. Kamm, G. N. et al. Fermi surface, effective masses, and Dingle temperatures of ZrTe5, as derived from the Shubnikov–de Haas effect. Phys. Rev. B 31, 7617 (1985).
26. Scanlon, D. O. et al. Controlling Bulk Conductivity in Topological Insulators: Key Role of Anti-Site Defects. Adv. Mater. 24, 2154–2158 (2012).
27. Iizumi, M. et al. Shubnikov–de Haas oscillations and Fermi surfaces in transition-metal pentatellurides ZrTe5 and HfTe5. J. Phys. C: Solid State Phys. 20, 3691–3705 (1987).
28. Analytis, J. G. et al. Bulk Fermi surface coexistence with Dirac surface state in Bi2Se3. A comparison of photoemission and Shubnikov–de Haas measurements. Phys. Rev. B 81, 205407 (2010).
29. Chen, J. et al. Tunable surface conductivity in Bi2Se3 revealed in diffusive electron transport. Phys. Rev. B 83, 241304 (2011).
30. Analytis, J. G. et al. Two-dimensional surface state in the quantum limit of a topological insulator. Nature Phys. 6, 960–964 (2010).
31. Xiu, F. et al. Manipulating surface states in topological insulator nanoribbons. Nature Nanotech. 6, 216–221 (2011).
32. Peng, H. et al. Aharonov–Bohm interference in topological insulator nanoribbons. Nature Mater. 9, 225–229 (2010).
33. Qu, D. X., Hor, Y. S., Xiong, J., Cava, R. J. & Ong, N. P. Crystal growth of BiTe6 on single-crystal substrate Sb2Te3 by molecular beam epitaxy. Science 329, 821–824 (2010).
34. Eto, K., Ren, Z., Taskin, A. A., Segawa, K. & Ando, Y. Angular-dependent oscillations of the magnetoresistance in Bi2Se3 due to the three-dimensional bulk Fermi surface. Phys. Rev. B 81, 195309 (2010).
35. Wang, L. X., Li, C. Z., Yu, D. P. & Liao, Z. M. Aharonov–Bohm oscillations in Dirac semimetal Cd3As2 nanowires. Nature Commun. 7, 10769 (2016).
36. Murakawa, H. et al. Detection of Berry’s phase in a Bulk Rashba semiconductors. Science 342, 1490–1493 (2013).
37. Taskin, A. A. & Ando, Y. Berry phase of nonideal Dirac fermions in topological insulators. Phys. Rev. B 84, 035301 (2011).
38. Wiendlocha, B., Resonant Levels, Vacancies, and Doping in Bi2Se3, Bi2Te3, and Bi2Se3–Bi2Te3 Tetradymites. J. Electron. Mater. 45, 3515–3531 (2016).
39. Chen, R. Y. et al. Optical spectroscopy study of the three-dimensional Dirac semimetal ZrTe5. Phys. Rev. B 92, 075107 (2015).
40. Taskin, A. A. & Ando, Y. Quantum oscillations in a topological insulator Bi1−xSex. Phys. Rev. B 80, 085303 (2009).
41. Aharonov, Y. & Bohm, D. Significance of Electromagnetic Potentials in the Quantum Theory. Phys. Rev. 115, 485 (1959).
42. Hong, S. S., Zhang, Y., Cha, J. I., Qi, X. L. & Cui, Y. One-Dimensional Helical Transport in Topological Insulator Nanowire Interferometers. Nano Lett. 14, 2815–2821 (2014).
43. Li, Z. et al. Two-dimensional universal conductance fluctuations and the electron-phonon interaction of surface states in Bi2Te5Se microflakes. Sci. Rep. 2, 595 (2012).

Acknowledgements

This work is supported by the National Key Research and Development Program of China (No. 2016YFA0308003, 2017YFA0206304), the National Natural Science Foundation of China (Nos 61474061, 61574079, 61274102, 11574137, 11774160), Jiangsu Shuangchuang Program, the Natural Science Foundation of Jiangsu Province (No. BK20140054) and UK EPSRC (EP/S010246/1). Pei Yang and Wei Wang contributed equally to this work.

Author Contributions

Pei Yang fabricated the devices, prepared Fig. 1a,b, Fig. 2c,d, Fig. 3 and Fig. 4 and wrote the main manuscript text with the help from Wei Wang and Liang He. Wei Wang synthesized the ZrTe5 NWs, carried out low-temperature transport measurements and XRD pattern. Xiaqian Zhang helped with the low-temperature transport measurements and carried out the TEM measurement. Kejie Wang prepared Fig. 1c and Fig. 2a. Liang He contributed to the analysis and paper revise. Wenqing Liu also revised the manuscript. Yongbing Xu reviewed the manuscript, too. Pei Yang and Wei Wang contributed equally to this work.

Additional Information

Competing Interests: The authors declare no competing interests.

Publisher’s note: Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.