Tuning the electrical transport of type II Weyl semimetal WTe$_2$ nanodevices by Ga$^+$ ion implantation

Dongzhi Fu$^1$, Bingwen Zhang$^1$, Xingchen Pan$^1$, Fucong Fei$^1$, Yongda Chen$^2$, Ming Gao$^2$, Shuyi Wu$^3$, Jian He$^1$, Zhanbin Bai$^3$, Yiming Pan$^4$, Qinfang Zhang$^3$, Xuefeng Wang$^2$, Xinglong Wu$^3$ & Fengqi Song$^1$

Here we introduce lattice defects in WTe$_2$ by Ga$^+$ implantation (GI), and study the effects of defects on the transport properties and electronic structures of the samples. Theoretical calculation shows that Te Frenkel defects is the dominant defect type, and Raman characterization results agree with this. Electrical transport measurements show that, after GI, significant changes are observed in magnetoresistance and Hall resistance. The classical two-band model analysis shows that both electron and hole concentration are significantly reduced. According to the calculated results, ion implantation leads to significant changes in the band structure and the Fermi surface of the WTe$_2$. Our results indicate that defect engineering is an effective route of controlling the electronic properties of WTe$_2$ devices.

Since the discovery of graphene$^{1-3}$ and topological insulators$^{4-7}$, the study of various new topological materials has become a hot spot. Recent years Weyl semimetals (WSMs), first implemented in the TaAs family$^{8-11}$, have aroused a wide range of interest due to their unique band structures and transport properties. Many prediction including Weyl point, Weyl cone, Fermi arc and chirality anomaly have been demonstrated by the experiments including angle-resolved photoemission spectroscopy$^{9,10,12-14}$, negative longitudinal magnetoresistance$^{15-17}$. Recently, a type of WSM, type II WSM, is proposed and verified in WTe$_2$$^{18-22}$, MoTe$_2$$^{23-27}$ and Mo$_x$W$_{1-x}$Te$_2$$^{28-30}$, etc. It is characterized by a few sets of heavily titled Weyl cones, whose Weyl points appear at the contact of electron and hole pockets. Due to the unique properties of the band, the theory predicts that there are many unique properties different from those of type I: momentum space Klein tunneling$^{31}$, field-selective chiral anomaly$^{32}$ and intrinsic anomalous Hall Effect$^{33}$, which becomes a good platform with intense research efforts.

For WTe$_2$, an extremely large positive magnetoresistance (XMR) has been observed, which has a value of 13,000,000% up to 60T at 0.53K with current along a- axis and magnetic field parallel to c-axis$^{34}$. Monolayer WTe$_2$ with 1T’ structure is predicted theoretically to be quantum spin Hall insulator$^{35}$ and edge conduction, which is confirmed recently$^{36}$. Pressure-induced dome-shaped superconductivity is reported in WTe$_2$ with a maximum critical temperature (Tc) of 7 K at around 16.8 GPa$^{37,38}$. Recently WTe$_2$ is predicted to be type II WSM$^{18}$ and chiral anomaly induced negative magnetoresistance is observed in WTe$_2$ devices of thin film and bulk materials$^{39-41}$. However, theoretical and experimental studies on lattice defects and their kinetics of WTe$_2$ are still absent. Lattice defects have a significant impact on the properties of WTe$_2$, so research on this is urgently needed. As the first theoretical candidate of type II WSM, WTe$_2$ have four pairs of Weyl points which are believed to be 50–60meV higher than the Fermi level of the pristine solid$^{42}$. Such a subtle control can often be tackled by the defect engineering$^{12,43}$.

1National Laboratory of Solid State Microstructures, Collaborative Innovation Center of Advanced Microstructures, and College of Physics, Nanjing University, Nanjing, 210093, P. R. China. 2National Laboratory of Solid State Microstructures, Collaborative Innovation Center of Advanced Microstructures, and School of Electronic Science and Engineering, Nanjing University, Nanjing, 210093, P. R. China. 3Key Laboratory for Advanced Technology in Environmental Protection of Jiangsu Province, Yancheng Institute of Technology, Yancheng, 224051, P. R. China. Dongzhi Fu and Bingwen Zhang contributed equally to this work. Correspondence and requests for materials should be addressed to Q.Z. (email: qfangzhang@gmail.com) or F.S. (email: songfengqi@nju.edu.cn)
Ion implantation is an important way to introduce lattice defects in crystals, and has been widely used in the past research. Here we report that, GI is carried out on WTe$_2$ nanodevices of thin flakes. Raman characterization, transport measurement and theoretical calculation determine the dominant defects and its effect on the device transport.

Results and Discussion

WTe$_2$ is a member of laminar transition metal dichalcogenides (TMDs). Unlike other TMDs, the Td phase is the naturally stable structure of WTe$_2$. Due to the weak Van der Waals force between different layers, WTe$_2$ can be easily exfoliated into films with atomic thickness. In our work, WTe$_2$ thin flakes are obtained by mechanical cleavage of strip-like crystals onto 285 nm-SiO$_2$/Si substrates. Figure 1a shows a typical optical image of the WTe$_2$ devices with Hall-bar geometry, and the thickness of the thin flake is $\sim$25.3 nm, determined from the height profile of the film along the black line plotted in Fig. 1b. Inset shows the original image of atomic force microscopy along the black line.

Here ion implantation is carried out in order to introduce structural defects in the 3D semimetal WTe$_2$. GI is carried out in a FEI Helios Nanolab 600i dual beam system with Ga$^+$ ion sources. A detailed description of ion implantation process is given in the method section. The candidate structural defects created by ion implantation include Frenkel defects and substitution defects, whose influence on transport properties and band structure will be elaborated in the following section. Here the software “The Stopping and Range of Ion in Matter” (SRIM) is used to simulate the process of GI. Figure 1c shows the simulation for 100 Ga$^+$ implantation into WTe$_2$ flakes. The simulated diagram was got by software “The Stopping and Range of Ion in Matter” (SRIM) with ion energy 30 keV. The red dots show the vacancies created by Ga$^+$, while the green dots are vacancies caused by recoiling W or Te atoms. (d) Ga$^+$ density profile of the WTe$_2$ film with GI. Ten thousand Ga$^+$ with ion energy 30 keV were used in SRIM to obtain the distribution curve.

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In order to determine the dominant type of structure defects, we characterize our samples by Raman spectroscopy. The calculated results about all 33 Raman vibrational modes in ref. are applied in our study. Figure 2a show
all the five Raman vibrational modes observed in sample A (~8.5 nm) with laser applied in the $z$ ($xx$) $z$ geometry. As shown in Fig. 2a, the first four modes ($A_2^4$, $A_1^9$, $A_1^8$, and $A_1^5$) are induced by the relative movements of Te atoms, while the $A_1^2$ mode is caused by the displacement between neighbouring W1 and W2 atoms$^{46}$. Among all the five modes, pure longitudinal and transverse optical modes are represented by $A_2^4$ and $A_1^5$ respectively$^{46}$.

Raman spectra of sample A with incident Laser along the c-axes at room temperature is shown in Fig. 2b. The green line show the experimental data for pure sample A (without GI and annealing), blue line represent the Raman spectra for sample A with GI (irradiated to total doses of 0.44 $\mu$C. cm$^{-2}$) and red line is the Raman spectra for sample A with GI and annealing. All the peak appearing in the range from 100~250 cm$^{-1}$ are analyzed by Lorentz fitting, and the results are shown in Table 1. On the basis of calculation results in ref. 46, five Raman vibrational modes are identified as shown in Fig. 2b. The $A_2^4$ ($\sim$111.1 cm$^{-1}$), $A_1^9$ ($\sim$116.7 cm$^{-1}$), $A_1^8$ ($\sim$133 cm$^{-1}$), $A_1^5$ ($\sim$163.5 cm$^{-1}$), and $A_1^2$ ($\sim$211.4 cm$^{-1}$) modes were allotted in sequence for pure sample A, which agrees with the results in ref. 46. In the meantime, the $A_2^4$ ($\sim$111 cm$^{-1}$), $A_1^9$ ($\sim$116.8 cm$^{-1}$), $A_1^8$ ($\sim$133.2 cm$^{-1}$), $A_1^5$ ($\sim$163.5 cm$^{-1}$), and $A_1^2$ ($\sim$211.8 cm$^{-1}$) modes were allotted sequentially for sample A with GI. The $A_2^4$ ($\sim$111.3 cm$^{-1}$), $A_1^9$ ($\sim$116.7 cm$^{-1}$), $A_1^8$ ($\sim$133.3 cm$^{-1}$), $A_1^5$ ($\sim$163.8 cm$^{-1}$), and $A_1^2$ ($\sim$212 cm$^{-1}$) modes were allotted sequentially for sample A with GI and annealing.

First of all, the peak positions almost show no change after GI and annealing. The weak changes in peak position are comparable with measuring error of the instrument. Furthermore, the full width at half maximum (FWHM) of the Raman peaks change significantly. GI increase the FWHM by introducing lattice defects, while annealing decrease the FWHM by repairing part of the defects and improve the quality of the samples. So the significant changes of the FWHM have confirmed the effect of “GI” and “annealing after GI” very well, which is consistent with the our expectation. Considering that no excess defect peak is observed, no major peak disappears.

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**Table 1.** Raman parameters comparison for pure sample A, sample A with GI and sample A with GI and annealing. All the parameters in the table are obtained by Lorentz fitting.

| Sample A Mode | Peak position/cm$^{-1}$ | Full width at half maximum/cm$^{-1}$ | Relative intensity I(Pi)/I(P5) |
|---------------|--------------------------|--------------------------------------|-------------------------------|
|               | Pure                     | With GI and annealing                | Pure                          | With GI                      | With GI and annealing |
| P1 $A_2^4$    | 111.1                    | 111                                  | 111.3                         | 2.95                         | 3.8                   | 3.55                 | 0.25795             | 0.18738             | 0.24337             |
| P2 $A_1^9$    | 116.7                    | 116.8                               | 117.1                         | 2.6                           | 3.17                  | 3.97                 | 0.11159             | 0.09701             | 0.1027              |
| P3 $A_1^8$    | 133                      | 133.2                               | 133.3                         | 3.31                         | 4.49                  | 4.15                 | 0.22329             | 0.14206             | 0.17697             |
| P4 $A_1^5$    | 163.5                    | 163.5                               | 163.8                         | 2.62                         | 3.65                  | 3.18                 | 0.59845             | 0.4483              | 0.56087             |
| P5 $A_1^2$    | 211.4                    | 211.8                               | 212                           | 4.02                         | 4.66                  | 4.68                 | 1                  | 1                  | 1                   |

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**Figure 2.** Raman evidence for main defects in WTe$_2$ induced by GI. (a) Atomic displacements of Raman active modes in WTe$_2$. Here, “$\times$” and “.” indicate that the Te atoms move into and out of the $bc$ plane, respectively. (b) Raman spectra of WTe$_2$ flakes with incident laser along the c-axes at room temperature. The green, blue and red line show the Raman spectra for pure sample A, sample A with GI and sample A with GI and annealing, respectively.
Table 2. Parameters used for GI and that derived from the two-band model fitting. $n$, $p$, carrier density for electron and hole respectively; $n/p$, the ratio of carrier density; $\mu_e$, $\mu_h$, mobility for electron and hole respectively.

| Sample | Thickness (nm) | Operating voltage (kV) | Dose (μCcm$^{-2}$) | $n$10$^{19}$cm$^{-3}$ | $p$10$^{19}$cm$^{-3}$ | $n/p$ | $\mu_e$(cm$^2$V$^{-1}$s$^{-1}$) | $\mu_h$(cm$^2$V$^{-1}$s$^{-1}$) |
|--------|----------------|------------------------|---------------------|---------------------|---------------------|------|---------------------|---------------------|
| B      | 25.3           | 30                     | 2                   | 0.4                 | 4.460               | 1.750 | 4.339               | 1.723               | 1.028               | 1.016               | 2161                | 1018                | 1736                | 994                |
|        |                |                        |                     |                     |                     |      |                     |                     |                    |                     |                    |                    |                    |
Carrier concentration ratio is almost unchanged in sample B with GI, suggesting that electron-hole compensation is maintained. However sample D with thinner thickness exhibits breakdown of electron-hole compensation shown in Fig. S3 of supplementary information. Here the two band model is used to reproduce correctly the hall resistivity of sample B. Table 2 summarizes the evolution of the electron and hole concentration, mobility and carriers concentration ratio, respectively. The mobility for electron decline greatly from 2161 cm²V⁻¹s⁻¹ to 1018 cm²V⁻¹s⁻¹, and the mobility for hole decay from 1736 cm²V⁻¹s⁻¹ to 994 cm²V⁻¹s⁻¹, which stem from the emergence of a large number of scattering centers. As shown in the Table 2, after GI, both electron and hole densities decrease obviously, implying the change of band structure and Fermi level in WTe₂ flake. Interestingly, carrier concentration ratio is almost unchanged in sample B with GI, suggesting that electron-hole compensation is maintained. However sample D with thinner thickness exhibits breakdown of electron-hole compensation shown in Table S4 (supplementary information). So introduction of structure defects by ion implantation is an effective method to modulate the carrier density, especially in semimetal devices, which cannot be tuned effectively by traditional gating due to the limit of screen length.

In addition to Raman characterization and transport measurements, we also investigate the impact of GI on the band structure and Fermi surface of WTe₂. We performed our calculation by the Vienna Ab-initio Simulation Package (VASP) code52–55. The distribution of high symmetry points in the first Brillouin zone is shown in Fig. S4 in supplementary information. Figure 4a–e are the crystal structure diagrams of pure WTe₂ and all of the defect types we consider in this work, including Te/W atom Frenkel defects and substitution defects. At the same time, Fig. 4f–j are the corresponding band structure for pure WTe₂ and WTe₂ crystal with a certain concentration of specific defects. As shown in Fig. 4f–j, the red line indicates the current position of Fermi energy for WTe₂ crystal with a certain concentration of specific defects, while the blue line represents the position of the Fermi energy for the pure WTe₂ crystal. As can be seen from Fig. 4f–j, the introduction of a certain concentration of structure defects in pure WTe₂ crystal, will result in a significant change in the band structure. In addition, the location of the Fermi surface will be significantly changed. The introduction of a certain concentration of W atom Frenkel defect has little effect on the Fermi energy of the WTe₂ crystal, while the introduction of the remaining three defects leads to a significant lifting in Fermi energy. The changes in Fermi level caused by various crystal defects are recorded in Table S5 in supplementary information. Considering that GI can cause a decrease in carrier concentration, we can speculate that Te atom Frenkel defect as the main defect type, is likely to result in the reduction of density of state (DOS). However the effect of other types of defects on the DOS remains to be studied.

Conclusion
In summary, by GI, we have effectively introduced lattice defects in WTe₂ thin film devices. Through Raman analysis and formation energy calculation, Te Frenkel defect is identified as the dominant defects in WTe₂. Transport measurements help us analyze the effects of lattice defects. In particular, the two band model analysis shows a significant decrease in electron and hole concentration induced by Te Frenkel defects. By theoretical calculation, we know the effect of all the candidate defects on band structure and Fermi surface. Ion implantation is expected to become a new way to control the electronic properties for WTe₂ nanodevices, meantime this approach may also apply to thin film devices of other layered TMDs.

Figure 3. Effect of lattice defects on transport properties. (a) Temperature dependence of the resistance $R_{xx}$ in zero field for sample B. The green line represents the experimental data for pure sample B and the blue line for sample B with GI and annealing. (b and c) display the field dependence of MR and $\rho_{xy}$ at 2 K, respectively, with a magnetic field applied along the c-axis direction. The red dotted line in (c) represents the two band model fit for sample B.
Methods

Devices fabrication. WTe₂ thin flakes were obtained by mechanically exfoliating bulk single crystals onto a Si wafer covered with a 285-nm thick thermally grown SiO₂ layer. Optical microscopy and atomic force microscopy were used to characterize the quality and thickness of the thin flakes. Once thin flakes were identified on the substrate, conventional nano-fabrication techniques (photolithography, standard electron beam evaporation and lift off) were employed to attach electrical contacts (consisting of Ti/Au bilayers, typically 5/50 nm thick).

Ga⁺ ion implantation. GI were carried out in a FEI Helios Nanolab 600i dual beam system with Ga⁺ ion sources at room temperature. The size of the Ga⁺ ion beam spot was set to 25 um × 20 um which can cover all the channel areas of WTe₂ devices, with uniform beam current 2pA. Working voltage of dual beam system was set to 30kV which corresponds to a projection range of 13.7 nm. The duration of implantation was limited to 3s to avoid the serious degradation of transport property of WTe₂ devices.

Annealing. After the process of GI, annealing treatment was carried out on the devices to repair the most cascade damage originating from the ion collision. WTe₂ devices were annealed in a horizontal tube furnace equipped with a 1 inch diameter quartz tube. Argon(89%)/H₂(11%)/mixed gases were kept at a flow rate of 100 sccm with mechanical vacuum pump working. The tube furnace was heated from room temperature to 240 °C within 60 min. After maintaining at 240 °C for 120 min, the system was naturally cooled down to room temperature.

Raman characterization. Raman spectra were characterized from a micro-Raman spectrometer (LabRAM HR) with a 514.5 nm wavelength Ar⁺ laser as the excitation source. The laser spot size on samples was a few microns in diameter, and the incident laser power was below 1 mW, which did not damage the studied devices.

Transport measurements. Four-probe measurements of magnetoresistance and Hall resistivity were conducted in a commercial cryostat (Quantum Design PPMS-14).

Electronic structure calculation. We performed our calculation by the Vienna Ab-initio Simulation Package (VASP) code. The Kohn-Sham equation have been solved variationally in a plane wave basis set using the projector-augmented-wave (PAW) method. The exchange correlation energy was described by the functional of Perdew, Burke, and Ernzerhof (PBE) based on the generalized gradient approximation.
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**Author Contributions**

F.S. conceived the original idea and D.F. did all the research. X.P. synthesized the $\text{WTe}_2$. Y.C., M.G., S.W. and J.H. carried out Raman characterization, F.F. performed the Ga$^+$ implantation. D.F. fabricated and measured the $\text{WTe}_2$ devices with help from Z.B., Q.Z. and B.Z. carried out the theoretical calculation. F.S., D.F., Q.Z., B.Z., Y.P., X.W. and X.W. participated in the analysis of the data and discussed the results. D.F., F.S. and B.Z. wrote the paper, and all authors provided their feedback. All authors have read and have approved the manuscript.

**Additional Information**

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**Competing Interests:** The authors declare that they have no competing interests.

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