



monolayer graphene provides indisputable evidence for the existence of a long-range ferromagnetic order. More recently, the first experimental realization of a four-terminal spin field effect transistor (FET) via a separate semiconducting single-walled carbon nanotube (SWNT) was reported [27]. Two partially open SWNT segments can be obtained when the Mo electrode is deposited using a magnetron sputtering.

Magnetoresistance (MR) is defined as the ability of a material to change the value of its electrical resistance in an externally applied magnetic field. The MR typically originated from charge inhomogeneity and other disorder in materials. The study of magnetoresistive behavior of metals can go back to experiments by L. Kelvin and E. Hall in 1857 and 1880, respectively. In 1928–1929, Kapitza and Rutherford [28, 29] reported the magnetoresistive behavior of 37 materials. He observed large MR in bismuth, arsenic and graphite when the magnetic field reaches 30 T. MR exhibits parabolic in small fields and increases linearly in fields above several tesla. The common positive magnetoresistance occurs in metals. Anisotropic magnetoresistance (AMR) refers to the phenomenon that the resistivity of ferromagnetic materials changes with the angle between the magnetization and current direction. In addition, for ferromagnets, a characteristic butterfly loop will be observed when the magnetic field is changed for ferromagnetism in iron single crystals and ferromagnetic semiconductor (Ga, Mn)As [30, 31]. Therefore, magnetoresistance measurement is a direct and alternative method to detect and study magnetic order of Ta-MWNTs. Esquinazi and his colleagues [32] reported that highly oriented pyrolytic graphite (HOPG) samples exposed to irradiation show anisotropic AMR and negative MR with pronounced hysteresis loops, which demonstrated the ferromagnetism in HOPG after irradiated. Temperature is an important factor in the change of materials properties and also has a significant effect on the magnetoresistance of magnetic materials. As the temperature increases to the Curie temperature of ferromagnetic materials, long-range order is broken down. The Curie temperature of traditional dilute magnetic semiconductors and two-dimensional intrinsic ferromagnetic semiconductors are lower than room temperature [33, 34].

Ta is known for its paramagnetic nature, high melting point, excellent thermal stability and good electrical conductivity [35]. Its paramagnetic nature excludes the effect of electrode metal on the magnetoresistive signal of CNT. Wang *et al.* [36] showed that the sputtering process creates more defects on the surface of Ta-decorated CNTs. According to Lieb's theorem [37], these defective structures can induce magnetic moments in Ta-covered CNT [38, 39]. Temperature affects the magnitude and direction of the magnetic moment [40]. As the temperature increases, the magnetic moment decreases and the direction becomes more random, which can be reflected by the

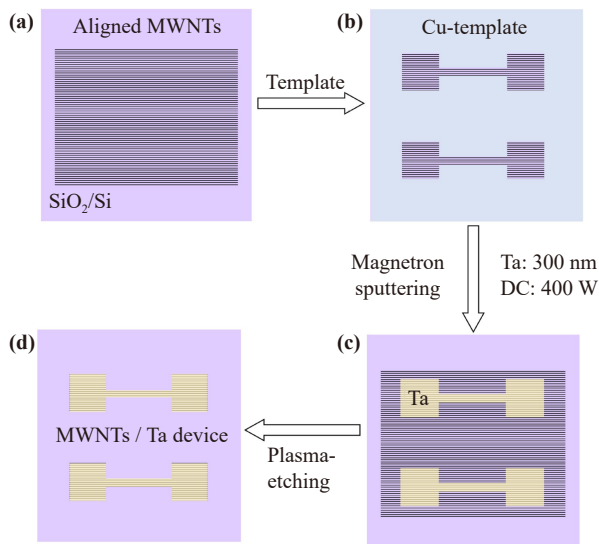
magnetoresistance behavior. Therefore, understanding the temperature-dependent behavior of magnetoresistance in MWNTs covered with Ta is essential for optimizing their performance in such applications.

In this study, we present our experimental procedures and describe the MWNTs/Ta device fabrication and magnetoresistance measurements performed under controlled temperatures using a Physical Property Measurement System (PPMS-ANC300) and four terminal techniques. We then elucidate the physical processes that contribute to magnetoresistance behavior by analyzing observed trends as a function of temperature. Our discussion focuses on positive and negative magnetoresistance separately and provides a comprehensive overview of how these phenomena evolve over a wide range of temperatures. This research has the potential to open up new avenues for the development of next-generation spintronic technologies with improved efficiency and functionality.

## 2 Experimental

The method used to fabricate the aligned MWNT films on a SiO<sub>2</sub>/Si substrate was the spinning continuous yarns method [41–45]. To ensure accurate test results, the MWNT film was soaked in a hydrochloric acid solution with a mass fraction of about 14.6% for 24 hours to eliminate the influence of ferromagnetic catalysts before device fabrication [Fig. 1(a)]. We then constructed MWNTs/Ta devices using hard templates and magnetron sputtering.

First, a metal mask is applied to the MWNT films. The metal mask has a dumbbell-shaped hollow in the middle, as shown in Fig. 1(b). The center hollow has a minimum size of 50  $\mu\text{m}$ . Hard templates are directly used as a masking film to cover the sample to prevent the effect of residual glue on the performance of the device during exposure processes and allow several devices to be prepared at the same time. Second, magnetron sputtering (Lesker Lad 18) is used to deposit Ta metal onto MWNT films. The base pressure and power are about  $1 \times 10^{-5}$  torr and 400 W, respectively, with a deposition time of approximately 250 s. After removal of the metal mask [Fig. 1(c)], argon plasma etching [Fig. 1(d)] is used to remove the excess carbon nanotubes from outside the device. The device is then ready for electrical testing. The Ta metal electrode is sufficiently thick to protect the MWNT films. Therefore, only the external MWNTs to the device are eliminated and the structure of the device remains not changed. Optical images of MWNTs/Ta and pure Ta devices are shown in Fig. 2(a). The devices exhibit a dumbbell-shaped configuration with the MWNTs running parallel to the Ta metal bars. Due to electrode expansion during the process of deposition, the minimum size at the center of the final device is approximately 100  $\mu\text{m}$ .

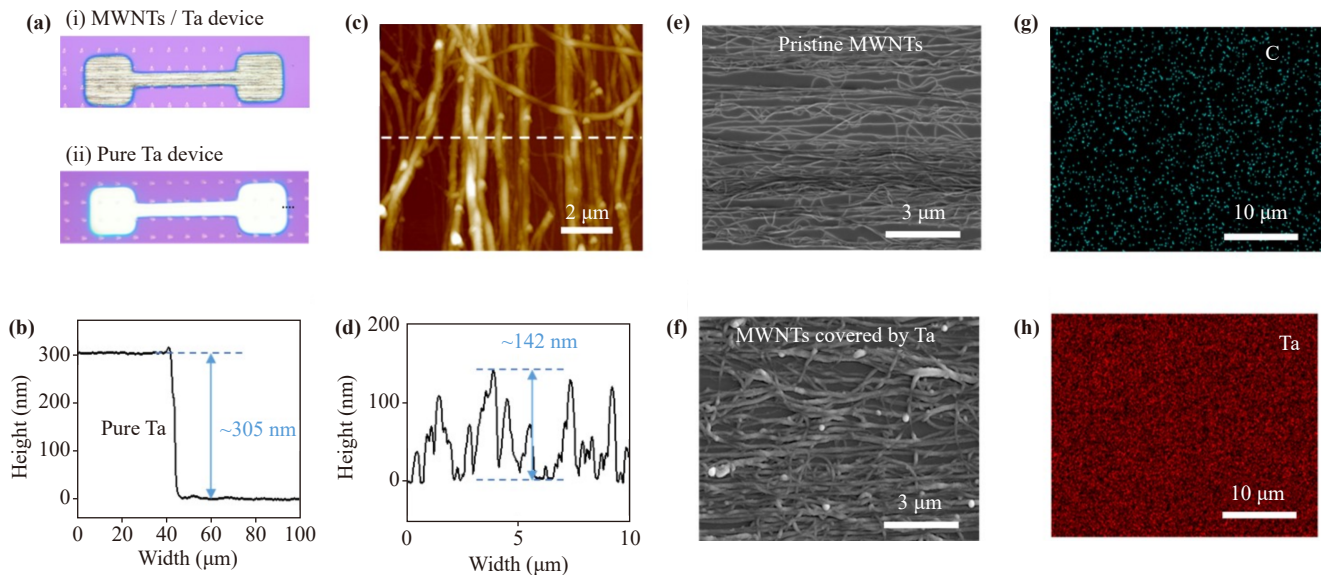


**Fig. 1** Fabrication of aligned MWNTs covered with tantalum. (a) Aligned MWNTs grown on silicon wafers. (b) A metal mask with a dumbbell is positioned onto MWNTs. (c) The metal tantalum (Ta) is deposited onto the MWNTs using magnetron sputtering. (d) Optical images of MWNTs/Ta bar devices.

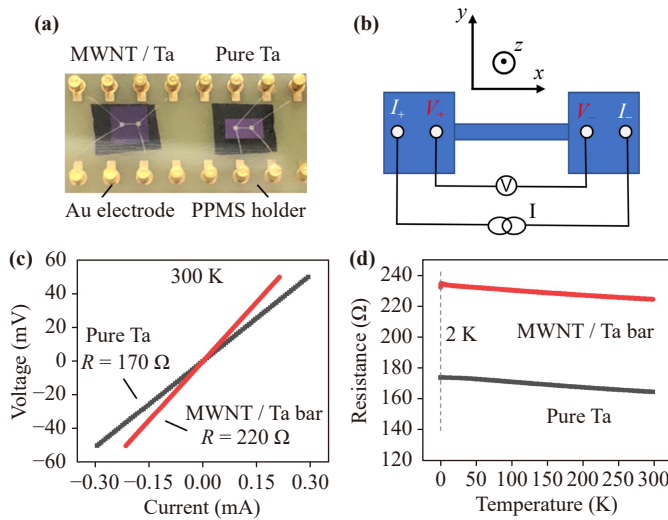
Using the same template and fabrication process, we also fabricated a pure Ta device. As shown in Fig. 2(b), the Ta thickness is  $\sim 305$  nm.

The morphology of the MWNTs/Ta was characterized using an atomic force microscope (AFM Multimode 8HR, Bruker), as shown in Fig. 2(c). The average height of the thinned MWNTs along the white dashed line is  $\sim 142$  nm, as shown in Fig. 2(d). This value is smaller than the tantalum metal electrode thickness (305 nm),

indicating that the MWNTs are squeezed under the Ta metal electrode. Images of pristine MWNTs and MWNTs covered with Ta metal obtained via Hitachi scanning electron microscopy (SEM) (SU8220) [Figs. 2(e, f)]. The diameter of MWNTs after magnetron sputtering is significantly wider than that of the pristine MWNTs, indicating that MWNTs are well covered with Ta metal. The morphology is used to understand the surface area and orientation of the MWNTs, which affects the adhesion and coverage of the tantalum coating. We performed AFM characterization and SEM characterization of Ta-covered MWNTs. Through AFM characterization, we found that the thickness of the Ta metal film is greater than the height fluctuation of the MWNTs, indicating that the MWNTs are completely covered by Ta metal. Through SEM characterization, it was found that the morphology of the Ta-covered MWNTs was significantly different from that of the pristine MWNTs, and the diameter of the Ta-covered MWNTs seemed to increase significantly, indicating that Ta was perfectly covered on the MWNTs. Then we used EDX (SEM) to characterize the element distribution of Ta-covered MWNTs device. The distribution of carbon and Ta elements was observed in the EDX mapping [Figs. 2(g, h)]. However, the distribution of iron elements was not observed, which demonstrates that there are few ferromagnetic impurities in Ta-covered MWNTs. Subsequently, to execute low temperature test, the pure Ta and MWNTs/Ta devices are bonded to the PPMS holder [Fig. 3(a)]. A four-terminal method is utilized for resistance testing due to the elimination of contact resistance. Figure 3(b) depicts the schematic drawing of the four-terminal method in this work. The outer ends of the device act as  $I_+$  and  $I_-$ ,



**Fig. 2** Characterization of aligned MWNTs covered with Ta. (a) Optical images of (i) MWNTs/Ta device, (ii) pure Ta device, and (b) the height profile of Ta device. (c) AFM image of aligned MWNTs/Ta. (d) MWNTs/Ta height profile. (e, f) SEM image of pristine MWNTs and aligned MWNTs/Ta. (g, h) The elements distribution of Ta-covered MWNTs.



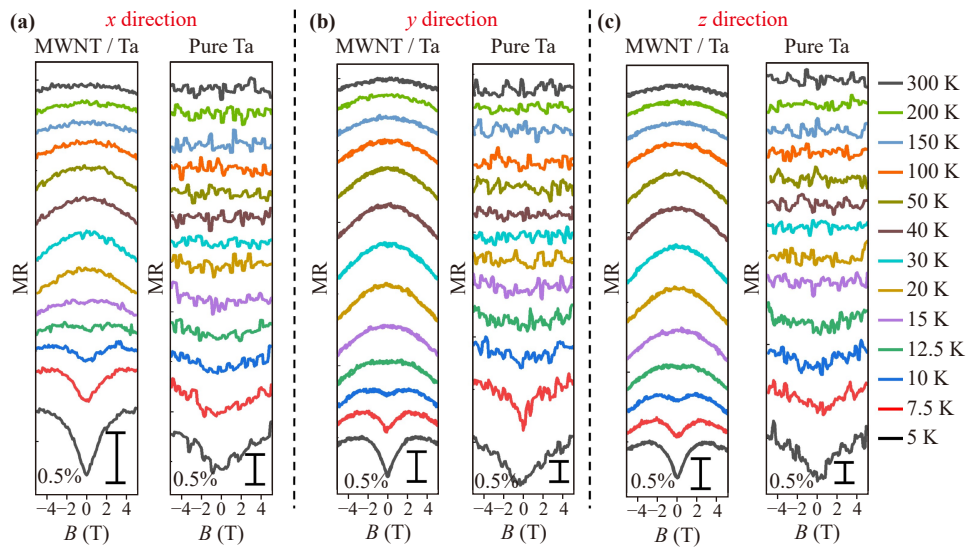
**Fig. 3** Bar structures and transport properties of MWNTs/Ta and Ta bar devices. (a) Photograph of MWNTs/Ta bar (left) and Ta bar (right) devices. (b) The schematic drawing of the four-terminal method. (c)  $I$ - $V$  characteristics of the devices in (a). (d) Resistance versus temperature of the devices in (a).

and the inner ends are used to measure the voltage difference ( $V_+ - V_-$ ). Finally, the resistance is obtained by Ohm's law. We conducted measurements of  $I_{ds}$  to achieve representative current-voltage ( $I$ - $V$ ) characteristic graphs of the Ta device and MWNTs/Ta device, which can be seen in Fig. 3(c). The device current varied linearly with voltage, and the resistance was 170 and 220  $\Omega$  for the Ta device and the MWNTs/Ta device, respectively. Figure 3(d) illustrates the variation in resistance as the temperature decreases from 300 to 2 K. The increase in resistances of the Ta device and Ta-covered MWNTs with decreasing temperature is mainly attributed to weak localization and electron-electron interactions in the Ta metal.

MR effect is a change in the material's resistance when exposed to an external magnetic field. We have carried out magnetoresistance tests on both pure Ta device and MWNTs/Ta device using the PPMS system. The transport performance of these devices in this work is measured in the temperature range of 5–300 K and scanning of magnetic fields from  $-5$  to  $5$  T. We express the direction of the magnetic field out-of-plane, perpendicular to the substrate, which was denoted by  $z$ . The direction of the magnetic field in-plane, parallel to the aligned MWNTs, was denoted by  $x$ . Finally, the direction of the magnetic field, in-plane, perpendicular to the aligned MWNTs, was denoted by  $y$ . To investigate the influence of the magnetoresistance of the device at various temperatures and magnetic field directions during the test, the device resistance was measured continuously as a function of the magnetic field under electric current. Figure 4(a) shows the variation curves of the resistance

of the MWNTs/Ta device and pure Ta device under magnetic field scanning in the  $x$  direction ( $-5$  to  $5$  T). At 5 K, it can be seen that the resistance value of the MWNTs/Ta device gradually increases with increasing magnetic field strength and finally reaches the saturation value, and exhibits positive magnetoresistance. At 7.5 and 10 K, the resistance initially increases and then decreases as the magnetic field strength increases. As the temperature rises further (12.5 to 300 K), a negative magnetoresistive behavior occurs, which means the resistance decreases as the magnetic field strength increases. Therefore, in the MWNTs/Ta device, a transition from positive magnetoresistance to negative magnetoresistance is observed with increasing temperature, which shows "M" shaped magnetoresistance at around 7.5 K. This phenomenon has also been reported by Geim *et al.* [46]. For the screened graphene device, they observed the M-typing MR under a small magnetic field. As the temperature increases, the magnetoresistive behavior changes from "M" to positive magnetoresistive behavior, as shown in the red frame in the figure below. They attribute this M-shaped to Landau quantization and the electron-hole plasma. For Ta-covered MWNTs, as the temperature increases, the magnetoresistive behavior changes from positive magnetoresistive behavior to M-shaped to negative magnetoresistive behavior. However, the mechanism of temperature regulating the magnetoresistive behavior of Ta-covered MWNTs is not clear. In addition, the resistance of a pure Ta device has a positive magnetoresistance of 5 to 12.5 K. And it is independent of the magnetic field strength and maintains constant when temperature is higher than 12.5 K, which is significantly different from MWNTs/Ta device. This difference between the MWNTs/Ta device and the pure Ta device is mainly caused by the magnetic momentum of the partially unzipped MWNTs. The magnetic moment comes from the solid-solid chemical reaction between Ta and carbon atoms, which unzipped the SWNTs along the radial direction. Following the coating of MWNTs with Ta, the surface of MWNTs/Ta exhibited the formation of defect sites, which disrupted the regular arrangement of MWNT atoms. This resulted in alterations to the magnetic moments and an impact on the overall magnetic behavior, which is consistent with previous studies [47, 48]. When applying a magnetic field is applied in the  $y$  direction and  $z$  direction, similar behaviors are observed [Figs. 4(b) and (c)].

MR is defined as  $MR = \frac{R(B) - R(0)}{R(0)}$ , the device resistance under an external magnetic field  $B$  is denoted by  $R(B)$ , while  $R(0)$  represents the device resistance in the absence of an external magnetic field. As shown in Fig. 5, the variation trend of MR (MWNTs/Ta device) with temperature is summarized. At 5 K, the MR of the MWNTs/Ta device exhibits a positive magnetoresistance under 5 T in the  $x$ ,  $y$ , and  $z$  directions. The values of MR are about 0.3%, 0.36%, and 0.34%, respectively.

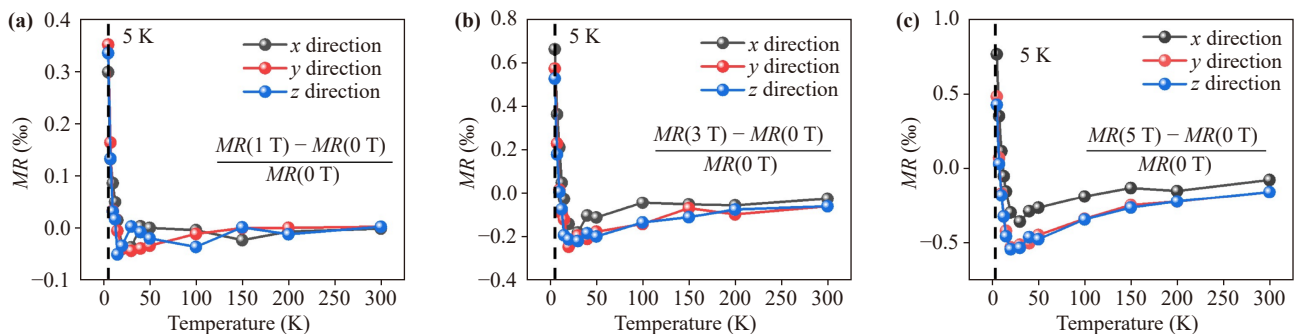


**Fig. 4** Magnetotransport properties for the MWNTs/Ta bar device and the pure Ta bar device were examined as a function of temperature in: (a)  $x$  direction, (b)  $y$  direction, and (c)  $z$  direction.

As the temperature increases, negative magnetoresistive behavior is observed and reaches a maximum of 15 K. With further increase in temperature, the negative magnetoresistive behavior slowly weakens and approaches zero.

Here, the negative magnetoresistance in the device made of aligned MWNTs covered with Ta is not due to the variation of magnetoresistance in Ta metal but due to other reasons. Chiral anomalies, the effect of ferromagnetic impurities and the magnetic moments at the edges of the MWNTs may be responsible for the influence of negative magnetoresistance of MWNTs/Ta devices at 10–300 K. First, Chiral anomalies in which electronic states of a particular chirality are not preserved when exposed to parallel magnetic and electric fields, are a common feature of negative magnetoresistance in materials and are usually found in highly conductive materials such as Weyl semimetals [49–52]. The magnetic field along the direction of the electric field is quite weak when the external magnetic field is parallel to the electric field. The chiral anomaly system exhibits a rapid

decrease in resistance with an increasing magnetic field, resulting in a negative magnetoresistance phenomenon. The negative magnetoresistance phenomenon observed in our experiments was not attributed to chiral anomalies. This phenomenon was observed regardless of the direction of the external magnetic field relative to the electric field. Second, the preparation of MWNTs by chemical vapor deposition requires the use of ferromagnetic catalysts. It cannot be excluded that the MWNTs used in our experiment contain trace amounts of ferromagnetic impurities, although subsequent transfer treatments were performed. Before device fabrication, we soaked silicon wafers with MWNTs in a solution of hydrochloric acid for 24 hours. The EDX mapping in Figs. 2(g) and (h) demonstrates that there are no ferromagnetic impurities in Ta-covered MWNTs. Based on the above discussion, it is suggested that the effect of negative magnetoresistance in the Ta-covered MWNTs device may be due to the magnetic moments at the open ends of the MWNTs. This is supported by experimental evidence of the magnetic moment and ferromagnetism of radial open-



**Fig. 5** Magnetoresistance (MR) behavior at different temperatures along different magnetic field directions: (a)  $x$  axis, (b)  $y$  axis, and (c)  $z$  axis of MWNTs/Ta bar device.

ended CNTs, which were applied to CNT spin devices and CNT film spin transistors [53].

### 3 Conclusion

We constructed MWNTs/Ta devices and pure Ta devices using template methods and magnetron sputtering. The device made from MWNTs/Ta had positive magnetoresistance at 5 K. Between 5 and 12.5 K, its magnetoresistance shifted from positive to negative and it displayed “M”-shaped behavior. In contrast to the test results for pure Ta devices, it demonstrated negative magnetoresistance between 12.5 and 300 K. The high-energy Ta atoms produced during magnetron sputtering reacted with MWNTs, resulting in magnetic moments at the radial opening of MWNTs, which made the MWNTs/Ta device less resistant to magnetoresistance. This work demonstrates the existence of magnetic moments at the radial openings of MWNTs and offers new perspectives on the ferromagnetism of carbon-based nanomaterials.

**Declarations** The authors declare that they have no competing interests and there are no conflicts.

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