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Ambipolar conduction in gated tungsten disulphide nanotube

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Devices based on transition metal dichalcogenide nanotubes hold great potential for electronic and opto-electronic applications. Herein, the electrical transport and photoresponse characteristics of a back-gate device with a channel made of a single tungsten disulfide (WS_2) nanotube are investigated as functions of electric stress, ambient pressure, and illumination. As a transistor, the device exhibits p-type conduction, which can be transformed into ambipolar conduction at a high drain–source voltage. Increasing ambient pressure enhances the p-type behaviour, while exposure to light has the opposite effect, enhancing n-type conduction. The ability to operate the device as either a p-type or n-type transistor makes it promising for complementary metal–oxide–semiconductor (CMOS) circuit applications. Light enhances the conductivity, allowing for further control and enabling the device to function as a photodetector with a photoresponsivity of about 50 mA W^{-1} and a broadband response in the visible range. The combination of voltage, pressure and light control paves the way for using the WS_2 nanotube transistor as a multifunctional device.

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1. Introduction

The 2004 isolation of graphene heralded a new era in materials science, sparking a wave of interest in two-dimensional (2D) materials.[1–6] Among these materials, layered transition metal dichalcogenides (TMDs) have emerged as key players in the field of nanotechnology. TMDs form a class of layered materials with the formula MX_2, where M is a transition metal from groups IV–VIB (Mo, Ti, Nb, W, etcetera) and X is a chalcogen atom (S, Se or Te).[7,8] These materials have garnered attention for their potential applications in electronics and opto-electronics due to their remarkable physical properties resulting from reduced dimensionality and crystal symmetry.[9] They have been extensively studied for their layer-dependent properties and optimal application performances.[10] The good gate control and down-scalability have positioned TMDs field-effect transistors (FETs) as promising structures for future electronic devices.[11] Additionally, the controlled synthesis of a few layered TMDs *via* vapor deposition techniques has paved the way for their integration into advanced microchip production.[12,13]

Among 2D TMDs, tungsten disulphide (WS_2) has garnered attention for its exciting properties and promising performance in various applications, including electronics and opto-electronics.[14] WS_2 exhibits strong spin–orbit coupling, band splitting, and high nonlinear susceptibility.[15–17] Structurally, WS_2 is a hexagonal layered compound with strong in-plane interactions and weak out-of-plane interactions, resulting in the limited amount of dangling bonds on the layers' edges and inert van der Waals surfaces.[18,19] This structural feature contributes to its stability and unique properties. The reduced effective electron mass in WS_2 has been predicted to result in the highest mobility and best performance in FETs among 2D TMDs, making it a promising candidate for advanced electronic devices.[20] Additionally, WS_2 has shown promise in opto-electronic applications due to its layer-tuneable bandgap and exotic valley physics, making it suitable for light–matter transduction.[21,22]

One-dimensional (1D) nanotubes (NTs) can offer several advantages over their 2D counterparts. They can enable transistors with smaller size and enhanced gate control through a gate-all-around configuration. More importantly, WS_2 nanotubes exhibit a bulk photovoltaic effect (BPVE) which can be exploited in a new generation of self-powered photodetectors. Indeed, moving from a two-dimensional monolayer to a nano-

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tube with polar properties, reduces the crystal symmetry beyond mere broken inversion symmetry of WS_2 and greatly enhances the BPVE.[23] Investigations into the potential of WS_2 NTs have showcased their electromechanical properties even beyond carbon NTs.[24,25] Indeed, they have been identified as a versatile class of materials with a wide range of potential applications. For instance, WS_2 NTs possess excellent electrochemical performance in lithium-ion batteries.[26] Additionally, WS_2 NTs exhibit high performance as photodetectors for visible and near-infrared light, positioning them as promising candidates for nanoscale optoelectronics.[27] Moreover, their significant bulk photovoltaic effect, particularly in the infrared region, could have implications for advanced photovoltaic applications.[23,28]

The synthesis of WS_2 NTs has been explored using various methods.[25,29,30] WS_2 NTs with small diameters and few walls are crucial for understanding their distinct characteristics.[31,32] Indeed, studies have explored the optical properties of WS_2 NTs, revealing that a decrease in NT diameter induces a redshift in photoluminescence, suggesting a narrowed band gap due to a curvature and strain effect.[33] In another study, the cathodoluminescence of WS_2 NTs with different diameters and amount of layers was investigated and compared to literature results. Here, the red shift of all types NTs *vs.* bulk WS_2 was attributed to the compatible effect between the quantum confinement (blue shift) in c-direction due to reduction of number of layers to single layer and strain promoted red shift.[34] Recent studies have also focused on the functionalization of tungsten disulfide (WS_2) NTs with various materials to enhance their properties for specific applications, such as fluorescence and conductivity.[35,36]

However, FETs based on individual WS_2 NT have not been extensively explored, compared to their 2D WS_2-based counterparts.[14,37] Notably, the pioneering work by R. Levi *et al.* in 2013 introduced the first FET[38] utilizing a single WS_2 NT, with limited subsequent studies reported. In this context, we have recently reported an individual multi-walled WS_2 NT which has been utilized as a channel of a back-gated FET, exhibiting p-type behaviour in ambient conditions and low bias, with a hole mobility $\mu_p \sim 1.4$ cm^2 V^{-1} s^{-1} and a subthreshold swing SS ~ 10 V dec^{-1}.[39]

In this work, we synthesized WS_2 NTs through a two-step reaction process and spread them onto a Si/SiO_2 substrate.[30] With the help of scanning electron microscopy (SEM), we determined the position of single NTs and contacted them using electron beam lithography. This device is a FET with p-type behaviour at low bias and ambient conditions, and conductance that can be tuned by ambient pressure. For consistency, all the data reported herein refer to the same device. However, we fabricated several devices in the same batch, which showed similar behaviour.[39]

Most importantly, we demonstrate that the device can switch from hole-dominant to electron-dominant conduction, *i.e.* it exhibits an ambipolar behaviour, at a drain–source voltage bias higher than about 1 V. Ambipolar devices, capable of functioning as both n-type and p-type transistors by adjusting the bias, offer flexibility in logic and analog circuit design.[40–42] Incorporating ambipolar transistors in CMOS (complementary metal–oxide–semiconductor) circuits enables the implementation of more complex logic functions per chip, thereby enhancing the computational capabilities of integrated circuits.

Furthermore, the device can be operated as a photodetector in the visible range, with a responsivity peak around 1.9 eV, correlated with WS_2 indirect band gap value. The control by electric bias, pressure and light can make WS_2 NT based transistors versatile devices in several applications.

2. Results and discussion

The schematic of the device under study is displayed in Fig. 1(a). A single WS_2 NT serves as the channel of the FET, where drain and source contacts are formed by titanium/gold (Ti/Au) metal pads. The channel current (I_d) can be modulated by the voltage applied to the gate terminal (V_{gs}) located at the back of the p-silicon substrate.

Transmission electron microscopy (TEM) was performed to check the structure of the NT and measure its radius. From the TEM image reported in Fig. 1b, we estimated the NT outer radius to be r = 17 nm, which is in the range of the average dimensions for NTs obtained through the same fabrication process (see Experimental section).[39] Here the interlayer distance in NTs' walls is of 0.63 nm, ~1.6% larger than that of bulk WS_2 (0.62 nm), which was assigned to strain in the rolled layers.[34]

Electrical characterization of the device was performed under dark and high vacuum conditions ($p = 1 \times 10^{-4}$ mbar). The I_d–V_{ds} output characteristic, *i.e.* the measurement of the channel current *versus* the applied drain–source voltage V_{ds}, at V_{gs} = 0 V, displayed in Fig. 1(c), reveals a slightly asymmetric Schottky behaviour which can be due to a small difference in the junction areas between NT and source/drain contacts.[43–45] The I_d–V_{gs} transfer characteristic at V_{ds} = 0.25 V in Fig. 1(d) reveals a hysteretic p-type conduction. The hysteretic behaviour reported in the transfer characteristic of the device is due to intrinsic defects and the presence of O_2 and H_2O on the NT surface that induce slow-time charge trapping, thus enlarging the difference between the forward and the reverse sweep curve.[46] From the transfer characteristic, we estimate the hole mobility using the following formula:

$$\mu_h = g_{m,max} \frac{L}{C_{ox} V_{ds}} \tag{1}$$

where $g_{m,max}$ is the maximum transconductance, L = 10 μm is the length of the NT, $C_{ox} = 13.5 \times 10^{-12}$ F m^{-1} is the oxide capacitance per unit length (the method for evaluating C_{ox} is reported elsewhere[47–50]). The analysis yields a hole mobility μ_p = 0.54 cm^2 V^{-1} s^{-1}, which corresponds to a hole density $p = \frac{J}{ev_d} = \frac{JL}{e\mu_p V_{ds}} = 2.2 \times 10^3\ \text{cm}^{-1}$, where J is the current density and v_d the carrier drift velocity.

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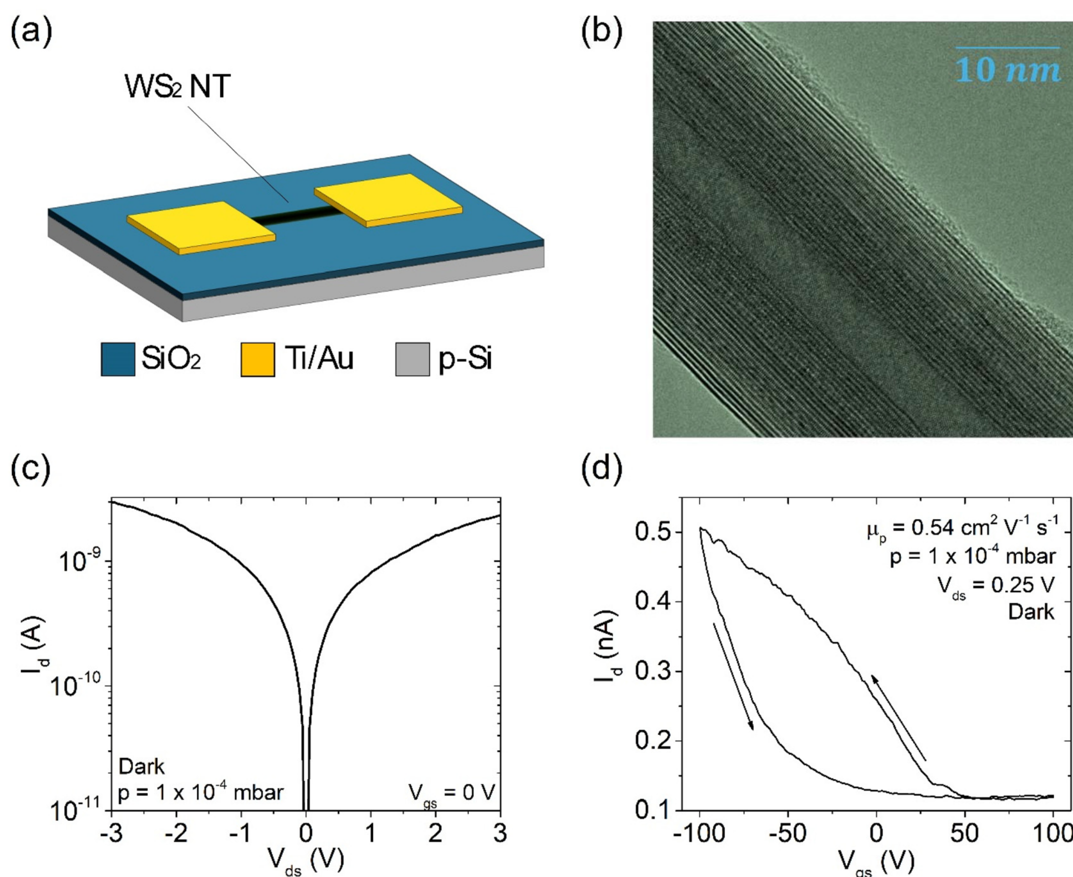

Fig. 1 (a) Schematic of the WS_2 NT device in back gate configuration. (b) TEM image of a typical WS_2 NT. (c) Output (I_d–V_{ds}) and (d) transfer (I_d–V_{gs}) characteristics of the WS_2 NT transistor in dark and high vacuum conditions.

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To complete the FET electrical characterization in high vacuum, we measured several I_d–V_{ds} characteristics at different applied V_{gs} (Fig. 2a). V_{gs} varies in the range ±100 V, with a step of 10 V. Fig. 2a shows different behaviours between forward and reverse branches. Indeed, at high positive V_{ds}, the drain current suddenly increases for V_{gs} > 30 V. This is clearly shown in the plot of Fig. 2b that reports the current at V_{ds} = ±3 V. The black curve in Fig. 2b reveals that the WS_2 NT transistor has an ambipolar behaviour at V_{ds} = +3 V, while the conduction is clearly dominated by holes at negative V_{ds} = −3 V, for which the transfer characteristic is p-type (red curve in Fig. 2b). We note that, at V_{ds} = +3 V, the *n*-branch at positive V_{gs} becomes

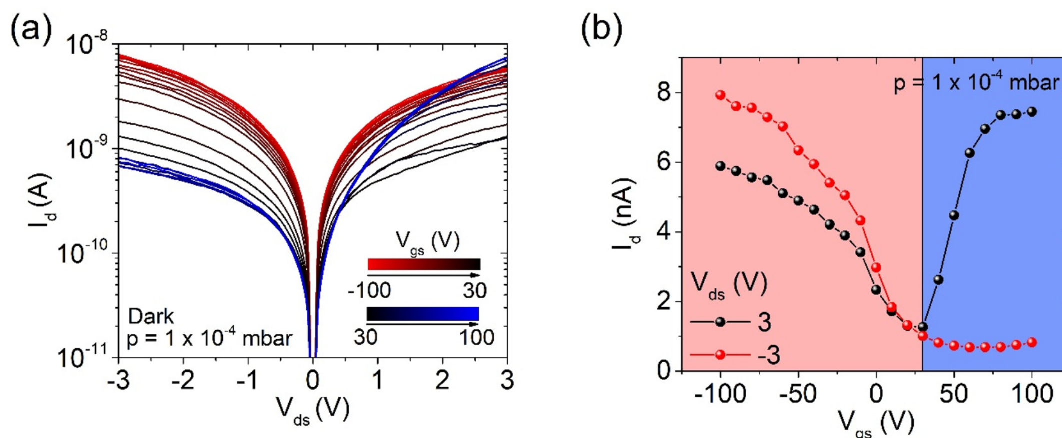

Fig. 2 (a) Output characteristics in dark and high vacuum conditions. (b) Current values extracted at ±3 V from data in (a).

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higher than the p-branch at negative V_{gs}, resulting in a complete switching from a p-type to a dominant n-type conduction.

With the aim to investigate the ambipolar conduction of the WS_2 NT FET, we further studied the transistor properties at positive V_{ds}. Fig. 3a reports the transfer characteristics of the device at different positive V_{ds}, from 0.25 V to 3.00 V, with a step of 0.25 V. The data show that, starting from a p-type device, the increase in V_{ds} activates electron conduction, thus enabling ambipolarity. Fig. 3b shows that, while hole mobility stays constant, the electron mobility increases with V_{ds}. Indeed, switching from p-type conduction to dominant n-type conduction occurs at about V_{ds} = 1.25 V, where electron mobility overcomes hole mobility.

Fig. 3c shows the transfer characteristics of the device measured at different V_{gs} ranges, from ±20 V to ±100 V, at a

Fig. 3 (a) Transfer characteristics at different V_{ds} in high vacuum. (b) Mobility extracted from $g_{m,max}$ evaluated in the reverse branch of the transfer characteristics in (a). (c) Transfer characteristics with different V_{gs} range in high vacuum. (d) Hysteresis *versus* applied V_{gs} range, extracted from data in (c). (e) Gate voltage V_{gs}^{min} and (f) minimum current I_d^{min} as a function of V_{gs}, for the forward and reverse sweep.

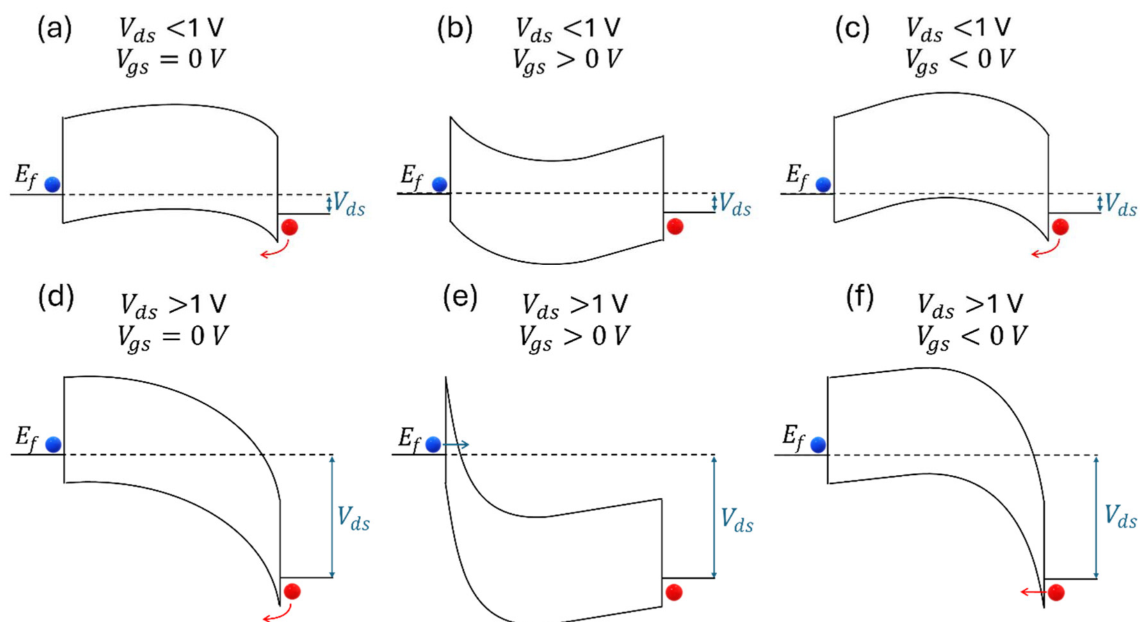

Fig. 4 Band diagram model at (a–c) low and (d–f) high V_{ds} voltage bias for different applied V_{gs}.

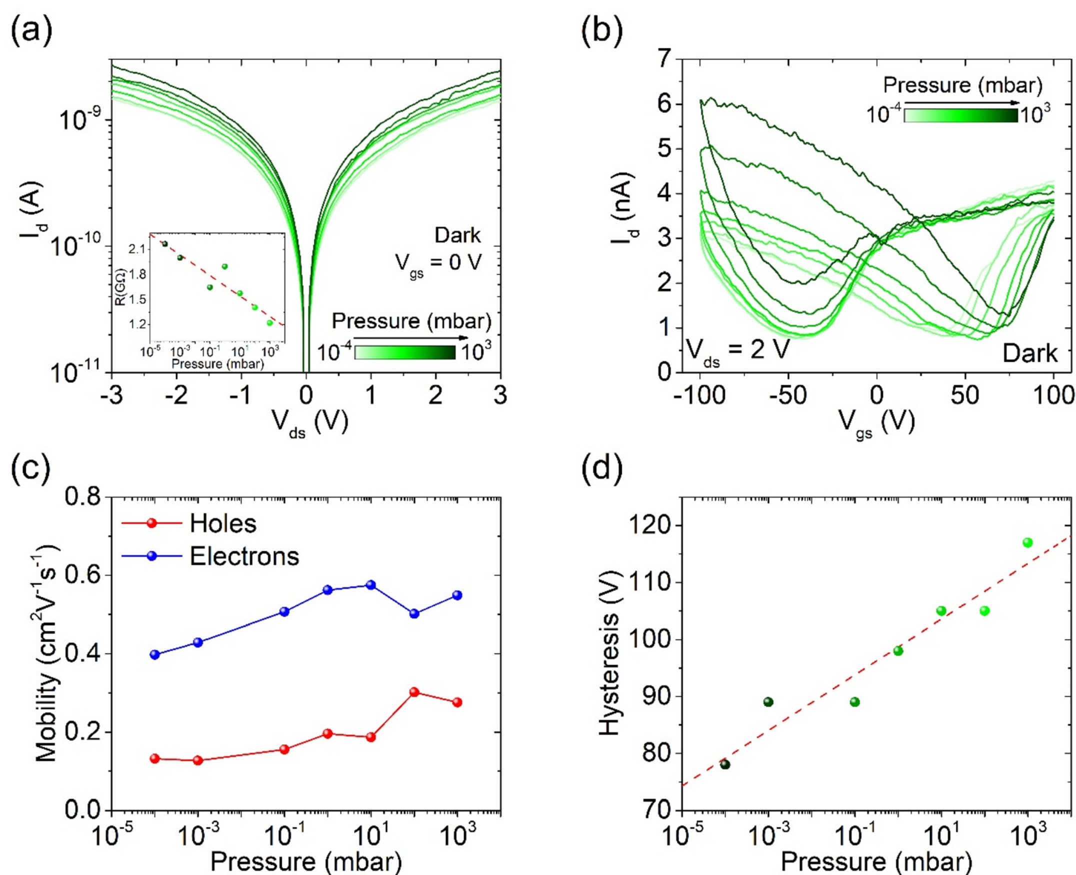

Fig. 5 (a) I–V curves at different pressures. Inset: resistance as a function of the pressure. (b) Transfer characteristics at different pressures. (c) Charge carrier mobility and (d) hysteresis width *versus* pressure.

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fixed V_{ds} = 2 V. To correctly evaluate the effect of the different V_{gs} ranges, we define the hysteresis H as the difference of V_{gs}^{min}, *i.e.* the voltage at which I_d is minimum (I_d^{min}), in reverse and forward voltage sweeps. H *versus* V_{gs} is plotted in Fig. 3d. The increase in the hysteresis with V_{gs} range is due to the increasing carrier trapping at the WS_2 NT/SiO_2 interface, due to the longer sweeping time.[41,51–53] The left-shift of V_{gs}^{min} during the forward sweep is stronger compared to the right-shift that occurs during the reverse sweep (Fig. 3e) and indicates that hole trapping is more effective than electron trapping.[54,55] Furthermore, the increasing V_{gs} range and measurement time also leads to an overall increase in current, as reported in Fig. 3c and highlighted in Fig. 3f, where the minimum values of the current are plotted *versus* V_{gs} range.

The observed behaviour can be explained by the band diagram model described in Fig. 4. WS_2 is a p-type semiconductor, hence the Fermi level (E_f) is close to the valence band, as demonstrate also by XPS.[25] Also, the work function of

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Fig. 6 (a) Output curves at different V_{gs} in dark and under illumination. (b) Transient photocurrent at different V_{gs} and (c) at different incident optical power. (d) Photocurrent and responsivity *versus* incident optical power. (e) Transfer characteristics at different V_{ds} in dark and under illumination. (f) Responsivity *versus* wavelength/energy of the incident laser beam.

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WS_2 is around 5.1 eV and is higher than the titanium work function, which is 4.33 eV.[25,56] These considerations lead to Fig. 4a, which shows the band diagram when low $V_{ds} < 1$ V and $V_{gs} = 0$ V are applied. In this scenario, a very low current, only due to the holes that can overcome the barrier at the drain contact, flows through the FET channel. When V_{gs} increases (Fig. 4b), the bands bend downwards, further blocking the migration of holes. In contrast, when a negative V_{gs} is applied, the bands bend upwards favouring the flow of holes through the channel (Fig. 4c). This explains the p-type conduction of the devices observed at low drain voltage.

On the other hand, when a high $V_{ds} > 1$ V is applied, the switching of V_{gs} from negative to positive values enables ambipolar conduction. Indeed, when $V_{gs} = 0$ V, holes can overcome the drain barrier, generating a p-type current (Fig. 4d). At positive V_{gs}, the bands bend downward, suppressing hole injection from the drain; however, the simultaneous thinning of the electron barrier at the source enables electron tunnelling, which is responsible for the emergence of a n-type current (Fig. 4e). Finally, at negative V_{gs}, hole conduction is favoured by the upward bending of the bands (Fig. 4f).

Increasing ambient pressure to standard conditions exposes the WS_2 NT to molecules such as oxygen and water. These molecules act as p-dopants when adsorbed on the surface of the NT. This results in an increase in hole density, *i.e.* of the channel conductance, and a slight change in the shape of the output and transfer characteristics of the device. The output (Fig. 5a) and transfer characteristics (Fig. 5b) measured at different pressures, from $p = 1 \times 10^{-4}$ mbar to standard ambient conditions ($p = 1 \times 10^{3}$ mbar), confirm the expected trend. Output characteristics reveal a resistance linear behaviour, which decreases with increasing pressure, as highlighted in the inset of Fig. 5a.

Electron and hole mobilities have been estimated and reported in Fig. 5c. The charge carrier mobilities are almost constant in the investigated range (that is, 7 orders of magnitude), hence they do not vary significantly with the pressure. Finally, Fig. 5d shows the enhancement of the hysteresis at high pressure, where the concentration of O_2 and H_2O molecules increases.

We also investigated the effect of light on the WS_2 NT FET properties, in high vacuum conditions. To this purpose, we exposed the NT to light from a supercontinuum laser. The laser beam has a nominal maximum output power P = 110 mW, which, scaled by the illuminated area of the NT and the laser spot ($A_{spot} = 25 \times 10^{-4}$ cm^2), results in an incident optical power P_{inc} = 80 nW. Fig. 6a shows the output curves in dark and under laser illumination at different applied V_{gs}. In particular, at negative V_{ds}, the effect of illumination is negligible at zero and negative high V_{gs}, while there is an appreciable photocurrent for high positive V_{gs}. This is due to the dominant p-type conduction for $V_{ds} < 0$, as already commented above. At positive V_{ds}, ambipolar conduction occurs and photoresponse varies for different V_{gs}. Fig. 6b reports the photocurrent I_{ph} (defined as the difference between light and dark current) peaks at different V_{gs}. Different photoresponse (V_{ds} = 2 V) occurs for different V_{gs}. The best performances are obtained for V_{gs} = 0 V, for which the highest response and the lower dark current are reported.

To further test the device as a photodetector, we changed the power of the laser beam while measuring the current flowing into the channel. The results are shown in Fig. 6c, where V_{ds} = 2 V and V_{gs} = 0 V, while P_{inc} varies from 15 to 75 nW. I_{ph} and the responsivity R (defined as $R = I_{ph}/P_{inc}$) are extracted from Fig. 6c and plotted against P_{inc} in Fig. 6d. Higher responsivity is reported at lower incident power, along with a photocurrent that increases with increasing P_{inc}. The increasing illumination power leads to an increase in photogenerated charge carriers, which corresponds to an increase in photocurrent. However, at high incident optical power, the enhanced carrier density leads to an increase in the scattering rate favouring electron–hole recombination and limiting the charge carrier mobility, thus resulting in a low responsivity.[57,58]

Moreover, transfer characteristics at different V_{ds} have been measured in the dark and under illumination (Fig. 6e). The incidence of the laser on the NT favours the desorbing of oxygen molecules from the surface, due to local heating, thus enhancing conduction by electrons, *i.e.* reversing the atmospheric p-doping effect.[59,60]

The responsivity as a function of the light wavelength is displayed in Fig. 6f. The peak at λ_{peak} = 643 nm (E_{peak} = 1.93 eV) falls within the range of energy band gaps of WS_2 NTs.[61,62]

3. Conclusion

We have investigated the electrical and optical properties of a single tungsten disulphide nanotube field effect transistor. We observed an ambipolar conduction that can be tuned by drain voltage, pressure and light. The transistor can switch from p-type to n-type conduction by increasing the drain–source voltage bias. We also demonstrated that the device can function as a photodetector with a good photoresponse in the visible range. The tuneable ambipolar conduction and photoconduction make the device promising for CMOS-integrated optoelectronic circuits.

4. Materials and methods

The synthesis of multiwall WS_2 nanotubes consisted in few steps.[30] Here, *via* a reduction–sulfurization process, the initially grown tungsten suboxide nanowhiskers were transformed into WS_2 nanotubes. The process is a one-pot synthesis, which means that both reactions (growth of oxide nanowhiskers and their sulfurization into WS_2 NTs) occurred in the same reactor, under identical conditions (same elevated temperature, same H_2S, H_2 and N_2 flows), and followed one another in a self-regulated manner. The nanowhiskers were synthesized through a series of intermediate reduction reactions from a blend of various WO_x ($2.83 \le x \le 3$) suboxide phases

and shapes. The process first included the reduction of the precursor into a volatile suboxide phase W_4O_{11} ($WO_{2.75}$), and its evaporation, further partial reduction of the $WO_{2.75}$ vapor into WO_2, and finally the "chemical" condensation of the vapor mixture into 1D nanocrystals giving the formation of a stable against additional reduction $W_{18}O_{49}$ suboxide phase. These suboxide nanowhiskers were then transformed into WS_2 nanotubes, starting from the surface and moving inward, towards the inner core through a slow diffusion-controlled sulfurization reaction.

The WS_2 nanotubes were then spread onto a Si/SiO_2 (tox = 500 nm) wafer, and their locations were determined using optical microscopy. Electrodes were created using electron beam lithography and electron beam evaporation of Ti/Au (20/120 nm) followed by a lift-off process in acetone.

The TEM sample characterization was performed with an FEI Tecnai G2 F20.

For the opto-electronic characterization, the device was tested in a Lakeshore probe-station with integrated optical fiber, connected to a Keithley Parameter Analyzer 4200-SCS. The pressure chamber was controlled by two external pumps (rotative and turbomolecular) and a needle valve.

The white laser source was a NKT Photonics Supercontinuum Laser (450–2400 nm) (SuperK COMPACT), with a nominal output power P = 110 mW and spectrum in the range 450–2400 nm.

4.1. Statistical analysis

All statistical analyses were performed using OriginLab software. We did not perform any pre-processing of the data, such as transformation, normalization, or evaluation of outliers.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of interest

There are no conflicts to declare.

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