Monolayer WS₂ crossed with an electro-spun PEDOT-PSS nano-ribbon: Fabricating a Schottky diode

Deliris N. Ortiz¹, Josee Vedrine¹, Nicholas J. Pinto^{1,*}, Carl H. Naylor², A.T. Charlie Johnson²

¹*Department of Physics and Electronics, University of Puerto Rico-Humacao, Humacao PR 00791*

²*Department of Physics and Astronomy, University of Pennsylvania, Philadelphia, PA 19104*

Abstract:

WS₂ and PEDOT-PSS were individually characterized with the goal of analyzing charge transport across a hetero-junction formed by these two materials. In thermal equilibrium electron flow from the WS₂ conduction band into the polymer LUMO level leads to band bending that creates a potential barrier preventing further current. The measured current-voltage (I_{DS} - V_{DS}) curve across the hetero-junction was non-linear and asymmetric similar to a diode, with a turn-on voltage of 1.4V and a rectification ratio of 12. The device I-V data were analyzed using the standard thermionic emission model of a Schottky junction and yielded an ideality parameter of 1.9 and a barrier height of 0.58 eV. This facile technique is the first report on a nano-diode fabricated using WS₂ and PEDOT-PSS, opening up the possibility of extending this work to include other layered transition metal dichalcogenides and conducting polymers.

Keywords: WS₂; PEDOT-PSS; Electrospinning; Diode

*Corresponding author: Nicholas J. Pinto

email: nicholas.pinto@upr.edu

1. Introduction

Successful large area growth of monolayer graphene[1-3] has raised interest in basic and applied research on other two dimensional electronic materials. They include layered transition metal dichalcogenides of the MX_2 type, where M is a transition metal from the IV – VI groups of the periodic table and X represents a chalcogen species like S, Se or Te[4,5]. These materials consist of a hexagonal layer of the metal atoms sandwiched between two layers of chalcogen atoms. MX_2 materials can crystalize in different forms, with the 2H phase of MoS_2 being the most widely studied. Recent work on the growth and characterization of 1T'- MoTe_2 shows evidence of it being a topological insulator[6]. Unsaturated d-orbitals of the transition metal imparts several of the interesting electronic properties to these materials, among these for example, relevant to this work, the *n*-type semiconducting behavior seen in tungsten disulphide (WS_2) is of particular interest for fabricating devices. Bulk WS_2 has an indirect band-gap in the range $1.3\text{eV} < \Delta E_g < 1.4\text{ eV}$ [7-9] while monolayer WS_2 has a direct band gap of $\sim 2\text{ eV}$ [8,10] and is stable under ordinary laboratory conditions. Air stable *n*-type materials are highly desirable and can be used in a wide range of electronic devices in combination with conducting polymers. Conducting polymers play an important role in organic electronics where low cost, flexibility, transparency and weight are important considerations among others[11-14]. The hole transporting poly(3,4-ethylenedioxythiophene) doped with (poly styrene sulfonic acid)-PEDOT-PSS is a commercially available polymer that is often used in organic electronic devices[15,16]. Using electrospinning, it is also easy to fabricate dry nano-fibers and nano-ribbons of this polymer which expands its use in nano-electronics[17,18]. The easiest device to fabricate is a hybrid organic/inorganic Schottky diode in which a hetero-junction of a hole transporting conducting polymer with a *n*-type inorganic semiconductor is formed[19]. Typically this construction has been achieved via electrochemical polymerization[20-23] or spin coating techniques[24], but this is “wet” process could result in undesirable chemical reactions with the inorganic semiconductor degrading device operation.

In this work, a straightforward technique of fabricating a WS₂ based Schottky nano-diode using PEDOT-PSS under normal laboratory conditions (room temperature, ambient pressure and illumination) is presented. We first electrically characterize monolayer WS₂ and PEDOT-PSS nano-ribbons individually with the goal of analyzing charge transport across a hetero-junction formed using these two materials. By simply crossing monolayer WS₂ with an electro-spun PEDOT-PSS nano-ribbon, in the absence of an external bias voltage, electrons from the conduction band of WS₂ move into the lower LUMO level of the polymer resulting in a barrier to charge flow. This is evidenced via the non-linear asymmetric current-voltage measurements made across the hetero-junction and is the first report on such a WS₂/PEDOT-PSS nano-device. The turn-on voltage was 1.4V and the rectification ratio was 12. A Schottky diode fabricated by us in the past using a polyaniline nanofiber and a n-doped Si substrate yielded a turn-on voltage in the range 0.4-0.6V and a rectification ratio of 160[25]. Using thicker WS₂ (where the band gap is reduced) films in this device would yield symmetric current-voltage curves, reducing the rectification ratio and making it difficult to define a turn-on voltage. Analyzing the results using the standard thermionic emission model of a Schottky junction yields a barrier height of 0.58 eV and an ideality parameter of 1.9. The complete exposure of the junction to the ambient makes this diode potentially useful as a gas or light sensor, while using a back gate bias to tune the carrier density could result in reversible manipulation of the barrier height. Diodes form the basic operating unit in AC signal rectification that powers all portable devices. Having one that is multifunctional is even better, and electro-spinning provides a novel method of fabricating such nano-diodes.

2. Materials and Methods

Mono- and multi-layer WS₂ flakes were synthesized via chemical vapor deposition (CVD) using a method developed in a previous report[26]. A 3.1mM solution of Ammonia Metatungstate (Sigma Aldrich, 358975) and 0.02g/mL sodium cholate is spun coated at 4000rpm on a Si/SiO₂ substrate. This

chip is placed in the center of a 1inch Lindberg Blue furnace and accompanied with a Sulfur chip placed upstream at a distance of 17cm from previous chip. The furnace is then ramped up to 800C at a ramp rate of 70C/min. Once at 800C, H₂ is introduced at 5sccm for 5min, and we believe that this initiates the WS₂ growth process. The H₂ is then stopped and the furnace is rapidly cooled to room temperature. Figure 1(a), (b) shows a scanning electron microscope (SEM) image of several WS₂ crystals grown via this method together with the electron diffraction spectra (EDS) of a selected portion of the image. WS₂ grows in a triangular shape consistent with its crystal structure[26]. The W and S peaks come from the crystals, while the Si and O peak is from the silicon oxide surface on which they were grown. Figure 1 (c), (d) shows an AFM image of a CVD grown WS₂ crystal together with the height profile along an edge. This crystal is one monolayer thick. After growth, the WS₂ crystals were transferred to a fresh doped Si/SiO₂ substrate via the use of a PMMA coated film and a KOH oxide etch as reported for a similar transfer of MoS₂[27]. This process reduces the probability of gate leakage currents (due to possible oxide damage during the growth process) when WS₂ is used in a field effect transistor (FET) configuration.

Commercial PEDOT-PSS was purchased from Baytron (Baytron P) as an aqueous dispersion, and nano-ribbons of widths in the range of a few microns and height of approximately 10nm were prepared using the electrospinning technique[18]. In order to cross WS₂ with a PEDOT-PSS nano-ribbon, the substrate with WS₂ crystals was rapidly passed in the path of the electro-spun jet in a downward sweeping action. This resulted in a few, air dried, PEDOT-PSS nano-ribbons sticking to the substrate, some of which intersect with the WS₂ crystals as shown in the optical microscope image of Figure 2(a) and some were isolated as they fell on regions of the substrate without any WS₂ crystals. Using an appropriate TEM grid as a shadow mask, Au or Ag electrodes were evaporated onto the substrate taking care to place the grid in such a way so as to cover the hetero-junction formed at the intersection of the WS₂ and PEDOT-PSS. The use of a TEM grid as a shadow mask eliminates any

need for harsh chemicals that are used in wet lithography and lift off procedures to make electrical contact pads on the device. Figure 2(b) shows the Au contact pads after thermal evaporation and removal of the grid. In all the measurements reported in this work, a Keithley electrometer model 6517A was used to supply the voltage and record the current. For three terminal field effect transistor measurements, an additional Keithley model 6487 pico ammeter/voltage source supplied the gate bias.

3. Results

Prior to electrical characterization of the crossed WS₂/PEDOT-PSS device, devices with individual WS₂ flakes or a single PEDOT-PSS nano-ribbon were fabricated and electrically characterized. Figure 3(a) shows a top view optical microscope image of a monolayer WS₂ crystal after evaporating 100nm Ag through a TEM grid that was used as a shadow mask. The dashed triangle circumscribes the WS₂ flake that is lying between two electrodes in this top contact configuration. The two black spots seen in this Figure are cured silver epoxy contacts that connect to Au wires (not seen) used for hard wiring the device, and the white squares are Ag contact pads. Figure 3(b) shows a schematic diagram of the WS₂ crystal connected as a FET with the external electrical circuit, while Figure 3(c) shows the drain-source current (I_{DS}) as a function of drain-source voltage (V_{DS}) of the device for various back gate (V_{GS}) biases taken in a vacuum of 10^{-2} Torr. For a fixed back gate bias, at low V_{DS} , the current increases linearly and shows signs of saturation at higher voltages as the channel gets pinched off, leading to a constant current flow, characteristic of FET's. Several devices were tested, and the channel current increased for increasing positive gate bias and confirm that WS₂ used in this work is an *n*-type semiconductor, although ambipolar field effects have also been observed for exfoliated WS₂[9]. At zero back gate bias there is no field induced charge in the channel, the WS₂ film conductivity was calculated to be 8.2×10^{-6} S/cm, a value that lies within the range of undoped semiconductors (for eg. amorphous Si). In the saturation regime of the I_{DS} - V_{DS} curve, the field effect

mobility (μ) can be calculated from the standard FET equation[28]:

$$I_{DS} = \frac{\mu W C_i}{2L} (V_{GS} - V_{TH})^2 \quad (1)$$

where L the channel length (15 μ m), W the channel width (20 μ m), C_i is the capacitance per unit area the 200nm SiO₂ film (1.725x10⁻⁸ F/cm²) and V_{TH} is the threshold voltage. The inset to Figure 3 shows a plot of $\sqrt{I_{DS}}$ vs. V_{GS} at a fixed $V_{DS} = 10$ V. The mobility calculated from the linear portion of this curve is 0.6 cm²/V-s and $V_{TH} = 10$ V after extrapolating the linear portion of the curve to V_{GS} at zero current. The on/off ratio was found to be 2x10⁵ at $V_{DS} = 10$ V and is slightly smaller compared to exfoliated WS₂ (~10⁶)[29]. The mobility however is better than that reported for CVD grown WS₂ (0.01 cm²/V-s)[30]. Defects resulting from the transfer of WS₂ to the new substrate lowers our measured mobility which we expect should be higher.

Figure 4 shows the current-voltage (I_{DS} - V_{DS}) characteristics of an isolated electro-spun PEDOT-PSS nano-ribbon taken in vacuum to avoid effects of humidity. The upper inset shows an optical micrograph of a typical electro-spun PEDOT-PSS nano-ribbon after Ag contact pads were thermally evaporated using a TEM grid as a shadow mask. When connected in a FET configuration, the I_{DS} - V_{DS} responses of the PEDOT-PSS nano-ribbons are linear, symmetric and the slope of the curve is unaffected by a back gate bias. This behavior is reasonable, since PEDOT-PSS is a conducting polymer with a high charge density that is unaffected by an external electric field and can be treated as an Ohmic conductor at low operating voltages. The conductivity (σ) can be calculated as follows:

$$\sigma = \frac{l}{RA} \quad (2)$$

where l is the length of the ribbon between the contacts, A is its cross-sectional area and R is the ribbon resistance calculated from the slope of the I-V plot of Figure 4. The conductivity calculated from the slope of the data is ~ 6 S/cm consistent for this conducting polymer[31, 32] and is due to the presence

of metallic islands formed by highly ordered polymer chains [33] dispersed in an amorphous matrix. 3-D variable range hopping is typically the favored charge transport mechanism in these highly conducting polymers[33].

Figure 5 shows the I_{DS} - V_{DS} characteristics of the device shown in Figure 2(b), where a monolayer WS_2 crystal is crossed with an isolated electro-spun PEDOT-PSS nano-ribbon. When the external voltage source positive terminal is connected to the PEDOT-PSS arm of the device and the negative terminal connected to WS_2 , the current is seen to increase linearly for increasing small positive applied voltage ($V < 1.0V$) and then increase more rapidly for larger voltages, similar to that observed in a diode under forward bias, while the current is suppressed under negative (reverse) bias. The device therefore turns on in the first quadrant for $V > 1.0V$ and is off in the third quadrant. When the external connections are reversed, the device turns on in the third quadrant as seen in Figure 5. This confirms that the non-linear asymmetric curve is due to a Schottky barrier formed at the intersection of the PEDOT-PSS nano-ribbon and WS_2 and is independent of any contact resistance with the external circuit. Since the mobility in WS_2 and the conductivity of PEDOT-PSS are comparable or higher than reported values[30,31], we believe that the contact resistance with the metal electrodes is not significant in the current device. The rectification ratio, which is the ratio of the forward bias current to the reverse bias current at $V=\pm 3V$ is 12 and is limited by the series resistance of each individual arm of the device and the barrier height. The turn on voltage obtained by extrapolating the linear portion of the forward bias curve to intercept the voltage axis at zero currents is 1.4V.

The formation of the Schottky barrier can be explained schematically with Figure 6. PEDOT-PSS is a p-doped conducting polymer with a band gap of ~ 1.6 eV[34] and no field effect response as seen in Figure 4. We assume that the Fermi level stays pinned midway between the HOMO and LUMO levels. Monolayer WS_2 is a *n*-type semiconductor as seen in Figure 3 with a band gap of ~ 2 eV, and hence its Fermi level is located closer to the conduction band. Figure 6(a) shows the band diagram

of PEDOT-PSS and WS₂ prior to the formation of the hetero-junction. Upon physical contact between the two, in thermal equilibrium, the Fermi energies of the two materials must match at the hetero-junction. This leads to band bending in order to establish a common Fermi level. During this process, electrons from WS₂ flow into the LUMO level of the polymer resulting in a barrier that prevents further flow of electrons into the polymer as seen in Figure 6(b). This barrier height is determined by the polymer work function and the surface states on WS₂[28].

In order to analyze the data, we assume the standard thermionic emission model of a Schottky junctions as follows[35]:

$$J = J_s [\exp \frac{qV}{nkT} - 1] \quad (3)$$

$$J_s = A^* T^2 \exp(\frac{-q\Phi_B}{kT}) \quad (4)$$

where J is the current density, J_s is the saturation current density, q is the electronic charge, k is the Boltzmann constant, T is the temperature, Φ_B is the barrier height and n is the ideality parameter (n = 1 is the ideal value) which takes into account corrections to the original simple model, e.g. image-force barrier lowering[28]. A* is the Richardson constant ($\frac{4\pi q m^* k^2}{h^3}$) is calculated to be 120 A/K²-cm² assuming m* is the electron mass.. At low bias there is a linear increase in the diode current as predicted by Eq. 3, while deviation from linearity at larger bias is generally related to Ohmic losses in the semiconductor. The inset to Figure 5 shows the semilogarithmic plot of the diode current as a function of applied voltage under forward bias conditions. Using the linear portion of this curve at low bias (V < 0.5V) yields a saturation current of 0.18 pA. The diode ideality parameter calculated from the slope of the curve as follows:

$$n = \frac{q}{kT} (\frac{\partial V}{\partial \ln J}) \quad (5)$$

is $n \sim 1.9$. Using these equations, we calculate the barrier height of 0.58 eV. A high ideality parameter could be due to recombination of hole and electrons at the interface or the presence of interface states[36] at the polymer/WS₂ interface. Further work using a back gate bias to tune the carrier density in WS₂ as seen in Figure 3, and thus manipulate the barrier height at the hetero-junction is planned in the near future. Since PEDOT-PSS nano-ribbons have been used to detect alcohol vapors[18], this also makes the diode an attractive candidate for a sensor thereby rendering it multifunctional.

4. Conclusions

Monolayer WS₂ was electrically characterized and shown to be a n-type semiconductor with charge mobility of 0.6 cm²/V-s and an on/off ratio of 2×10^5 , while the PEDOT-PSS nano-ribbon was highly conducting with no external field dependent charge transport. Combining these two materials in a unique configuration via electro-spinning yields a Schottky diode with a rectification ratio of 12 and a turn-on voltage of 1.4V. The diode current-voltage curve was analyzed using the standard thermionic emission model of a Schottky junction yielding an ideality parameter of 1.9 and a barrier height of 0.58 eV. This is the first report of a diode formed using monolayer WS₂ and PEDOT-PSS, and opens up the possibility of expanding this work to include other layered transition metal dichalcogenides of the MX₂ type and conducting polymers. In addition, it should be possible to tune the semiconductor carrier density via a back gate bias and thus reversibly change the barrier height, while the large surface to volume ratio of the film makes it potentially useful as a sensor. This device therefore could be multifunctional, capable of performing additional operations besides signal rectification as an active component in complex electronic circuits.

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Figure Captions:

Figure 1: (a) Top view scanning electron microscope (SEM) image of CVD grown WS₂ crystals on a doped Si/SiO₂ substrate (b) EDS spectrum of a section (yellow rectangle) of the image shown in Figure 1(a). (c) Atomic Force Microscope (AFM) image of a WS₂ crystal (d) AFM height profile of the edge of the crystal indicated by the black line in Figure 1(c). This crystal vertical height is ~0.9 nm or one monolayer thick.

Figure 2: (a) Top view optical microscope image of a PEDOT-PSS nano-ribbon intersecting WS₂ crystals that were grown and later transferred to a doped Si/SiO₂ substrate. The dark yellow triangles are the WS₂ crystals and the blue line is the PEDOT-PSS ribbon that was electro-spun over the substrate. This ribbon is seen to intersect two WS₂ crystals. The device formed by intersection of PEDOT-PSS with the larger WS₂ crystal was used in this work. (b) Magnified section of the dashed box seen in (a) after a TEM grid was used as a shadow mask and Au electrodes evaporated to make contact to WS₂ and PEDOT-PSS. The intersection of the ribbon with WS₂ was covered by the grid bar during evaporation shielding it from Au deposit. Current flow across the two Au electrodes must pass through this hetero-junction. The dashed triangle is the outline of the WS₂ crystal enclosed in the dashed box seen in (a). The image taken in (b) is rotated slightly clockwise compared to (a).

Figure 3: (a) Top view optical micrograph of a WS₂ crystal with electrical contact pads evaporated through a TEM grid. The dashed triangle indicates the location of a single crystal WS₂ flake. (b) Schematic of the WS₂ crystal connected in a FET configuration. (c) Drain-source current (I_{DS}) versus drain-source voltage (V_{DS}) for various back gate bias voltages (V_{GS}) taken in vacuum. The gate bias V_{GS} is shown to the right above each curve. Inset: Plot of $\sqrt{I_{DS}}$ vs. V_{GS} for fixed $V_{DS} = 10V$ and is used

for calculating the mobility and the threshold voltage.

Figure 4: Current-voltage (I_{DS} - V_{DS}) curve for an isolated electro-spun PEDOT-PSS nano-ribbon connected in a FET configuration in vacuum. There is no change in the slope of the curve for various back gate biases of -40V, 0V , 40V. Upper Inset: Optical micrograph an isolated electro-spun PEDOT-PSS nano-ribbon with Ag square contact pads evaporated through a TEM grid.

Figure 5: Current-voltage (I_{DS} - V_{DS}) characteristic curve measured in air for the Schottky diode shown in Figure 2(b) when the positive bias terminal was connected to the PEDOT-PSS arm and the negative to the WS_2 arm of the device ($\circ$), and when these external connections are reversed the diode turns on in the third quadrant ($\circ$). The inset above presents a semi-logarithmic plot of the forward bias current as a function of forward bias voltage. I_s is the reverse bias saturation current. The slope of the line is used to calculate the diode ideality parameter and I_s is used to calculate the barrier height.

Figure 6: Schematic band diagrams showing the formation of the hetero-junction when WS_2 is crossed with a PEDOT-PSS nano ribbon: (a) before crossing (b) after crossing. E_C , E_V and E_F are the conduction, valence and Fermi band edge energy levels of WS_2 and E_{FP} is the Fermi level of the PEDOT-PSS nano-ribbon. $\Delta E1$ and $\Delta E2$ are the energy band gaps for PEDOT-PSS and WS_2 respectively, ϕ_m and ϕ_s are the work functions of PEDOT-PSS and WS_2 , χ is the WS_2 electron affinity and V_b is the build-in potential. The HUMO and LUMO levels of the polymer are also indicated. After contact, in order to establish a common Fermi level at thermal equilibrium, the bands have to bend thereby setting up the Schottky barrier (indicated by the arrow) to the flow of electrons from WS_2 into the polymer.

Figure1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6