# 2D Materials

## Printed graphene/WS$_2$ battery-free wireless photosensor on papers

Ting Leng$^{1,2,7}$, Khaled Parvez$^{2,7}$, Kewen Pan$^1$, Junaid Ali$^2$, Daryl McManus$^{2,7}$, Kostya S Novoselov$^{3,4,5,6}$, Cinzia Casiraghi$^{2,8}$ and Zhirun Hu$^{1,4,6}$

$^1$ Department of Electrical and Electronic Engineering, University of Manchester, Manchester M13 9PL, United Kingdom
$^2$ Department of Chemistry, University of Manchester, Manchester M13 9PL, United Kingdom
$^3$ Department of Physics and Astronomy, University of Manchester, Manchester M13 9PL, United Kingdom
$^4$ National Institute of Graphene, Manchester M13 9PL, United Kingdom
$^5$ Centre for Advanced 2D Materials, National University of Singapore, 117546, Singapore
$^6$ Chongqing 2D Materials Institute, Liangjiang New Area, Chongqing, 400714, People’s Republic of China
$^7$ T Leng and K Parvez contribute equally to this work.
$^8$ E-mail: cinzia.casiraghi@manchester.ac.uk and z.hu@manchester.ac.uk

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## Abstract

Screen-printed graphene near field communication (NFC) tag antenna is integrated with inkjet-printed WS$_2$ photodetector on paper substrate to fabricate battery-free wireless photosensor. A sequential multi-stack printing is employed for the wireless photosensor fabrication: the NFC tag antenna is first screen-printed with graphene conductive ink and then the photodetector is inkjet-printed with transition metal dichalcogenides (TMDs) WS$_2$ ink as photoactive component. High responsivity and sensitivity are observed for the WS$_2$ photodetector, which acts as photoactive thermistor of the NFC sensor IC chip. The highly conductive graphene nanoflakes ink enables the screen-printed graphene NFC tag antenna to withdraw sufficient power wirelessly from the reader to power the sensor IC chip. This work demonstrates a prospective approach to manufacture 2D materials enabled electronics where the electronic circuits (normally having a large size) can be realized by mass production screen printing and the sensing component (normally having a small size) can be produced by inkjet printing, enabling low cost and simple fabrication methods, compatible with flexible substrates such as paper.

## 1. Introduction

The discovery of novel nanomaterials with outstanding properties and the development of simple fabrication techniques have strongly increased the demands in low cost, widespread, integrated printed electronics applications [1, 2]. The substrate is a decisive factor for the selection of the fabrication technologies and for the final application. In contrast to conventional substrates for printed electronics, such as glass and plastic, paper is one of the cheapest flexible substrates, which is also disposable and recyclable [3, 4]. Thus, a large amount of research is currently dedicated to devices fabricated on paper [5–10].

Graphene, a single layer of graphite, is the most researched 2-dimensional (2D) material due to its outstanding properties [11]. It can be produced in solution by liquid phase exfoliation (LPE) [12]. LPE can be also used to exfoliate other 2D materials, including conducting materials such as graphene, semiconducting materials such as transition metal dichalcogenides (TMDs) and insulating materials such as hexagonal boron nitride (h-BN) [13]. The exfoliated graphene and other 2D materials can be processed for ink formulation for screen and inkjet printing, enabling the development of fully-printable 2D materials electronics [14, 15]. Given that each 2D material has different properties, there is a very promising potential and prospect for the manufacturing of low-cost, large-scale printed functional devices [11–13, 16–19].

2D material inks are proven to possess unique electronic, optical and mechanical properties, which have demonstrated excellent compatibility with paper substrates using traditional printing techniques, such as screen printing and inkjet printing [10, 17, 20–23]. Among 2D materials, graphene’s high conductivity and strength make it an ideal material for the fabrication of low cost printed flexible interconnects,
electrodes and antennas [8–10, 16, 17, 20–23]. TMDs are layered compounds with an indirect band gap, which changes to direct gap upon exfoliating to single layer [24, 25]. Integrated with graphene electrodes, the complementary electronic properties of TMDs can be used to fabricate photodetectors [26, 27].

Most of the materials employed for printed electronics require rather harsh post-printing treatment like heating and acid treatments, which causes compatibility problems with many flexible substrates such as papers, textiles, plastic and elastomers [28]. Furthermore, the difficulties in recycling and the rapid accumulation of waste electronic devices in recent years have become insurmountable problems to the environment. The answer for both disposable and flexible electronics lies in using low-cost, biodegradable, and flexible substrates such as paper [3]. Furthermore, the porosity in paper allows fast absorption of the solvent, allowing the drop spacing to be minimized during printing for inkjet printing [21] and thick layer of ink deposition in screen printing [10]. The combination of the two printing techniques, where screen-printing can fabricate large area electronic circuits and inkjet printing can fabricate sensing components, has the potential to achieve fully-printed 2D materials electronics on paper.

In this work we use inkjet and screen printing 2D material inks made in our groups [10, 21, 22] to develop an all-printed, battery-free, wireless enabled 2D materials based photosensor on paper substrate. The photodetector is made by printing a heterostructure consisting of graphene as the electrodes and WS₂ as the photosensitive material connected to the NFC sensor IC chip as a photoactive thermistor. The IC chip is then connected to screen printed graphene NFC tag antenna for wireless power harvesting and data transmission. The highly conductive graphene ink enables the printed graphene NFC tag antenna to withdraw sufficient power wirelessly from the reader to power the sensor IC chip and transmit the data back to the reader. The wireless battery-free photosensor demonstrates high responsivity and sensitivity.

2. Materials preparation and methods

2.1. Inkjet printed WS₂ photodetector

Graphite (100 + mesh), WS₂ tungsten (IV) disulfide powder, (<2 µm, 99% grade) and 1-pyrenesulfonic acid sodium (PS1) salt (purity > 97.0%) were purchased from Sigma Aldrich. Both the graphene and WS₂ inks were formulated from their bulk counterparts via ultrasonic assisted liquid-phase exfoliation. Briefly, 1.5 g of WS₂ powder and 0.5 g of PS1 was dispersed in 500 ml of deionized water and sonicated at 600 W using a Hilsonic bath sonicator for 5 d at constant temperature of 15 °C. Afterwards, the dispersion was centrifuged at 903 g for 20 min using a Sigma 1–14 k refrigerated centrifuge to collect the supernatant and to remove unexfoliated WS₂. The collected supernatant has a proper monolayer percentage and flake size suitable for inkjet printing. The supernatant was then centrifuged again at 16 600 g for 60 min to remove
adjusted to 2 mg ml$^{-1}$.

Different geometrical parameters. The concentration of inkjet printable graphene ink was prepared using the whose composition has been described in [21]. The dispersed in the inkjet printable water-based solvent, excess PS1. The collected precipitate was then re-
dispersed in 400 ml of N-Methyl-2-pyrrolidone (NMP) (Sigma, anhydrous, purity: 99.5%) in a

Simulation results for printed graphene NFC tag with Table 1.

| No. of turns | Width $W$ (mm) | Spacing $S$ (mm) | Inductance $L$ ($\mu$H) | Resistance $R$ (Ω) | Quality factor |
|--------------|----------------|-----------------|------------------------|-----------------|---------------|
| 3            | 3              | 0.5             | 0.844                  | 125.980         | 0.571         |
| 3            | 1              | 0.791           | 121.780                | 0.553           |
| 3            | 2              | 0.688           | 115.500                | 0.508           |
| 4            | 0.5            | 0.710           | 89.640                 | 0.674           |
| 4            | 1              | 0.663           | 86.410                 | 0.654           |
| 4            | 2              | 0.579           | 81.610                 | 0.605           |
| 5            | 0.5            | 0.593           | 67.270                 | 0.752           |
| 5            | 1              | 0.551           | 65.020                 | 0.722           |
| 5            | 2              | 0.479           | 61.070                 | 0.669           |
| 4            | 2              | 0.911           | 288.310                | 0.269           |
| 2            | 1              | 0.913           | 269.940                | 0.288           |
| 2            | 2              | 0.965           | 243.930                | 0.267           |
| 3            | 0.5            | 0.772           | 191.680                | 0.343           |
| 3            | 1              | 0.764           | 177.350                | 0.367           |
| 3            | 2              | 0.651           | 156.530                | 0.354           |
| 4            | 0.5            | 0.647           | 138.780                | 0.397           |
| 4            | 1              | 0.620           | 124.260                | 0.425           |
| 4            | 2              | 0.480           | 109.370                | 0.374           |
| 5            | 1              | 0.553           | 200.160                | 0.128           |
| 1            | 0.960          | 659.680         | 0.124                  |
| 2            | 0.904          | 590.150         | 0.131                  |
| 2            | 0.5            | 1.456           | 352.140                | 0.352           |
| 2            | 1              | 1.341           | 321.530                | 0.355           |
| 2            | 2              | 1.072           | 278.540                | 0.328           |
| 3            | 0.5            | 1.242           | 220.850                | 0.479           |
| 3            | 1              | 1.066           | 199.990                | 0.454           |
| 3            | 2              | 0.796           | 168.630                | 0.402           |

excess PS1. The collected precipitate was then re-dispersed in the inkjet printable water-based solvent, whose composition has been described in [21]. The inkjet printable graphene ink was prepared using the same procedure described above. The concentration of inkjet printable graphene and WS$_2$ inks was carefully adjusted to 2 mg ml$^{-1}$. Photodetectors were printed with a Fujifilm Dimatix DMP-2800 piezoelectric inkjet printer through cartridges with a nozzle diameter of 21 µm. During the inkjet printing, neither the nozzles nor the substrate was heated. Samples with 40, 50, 60 passes of WS$_2$ were printed as photoactive layer, while the electrodes were printed with 20 passes of graphene ink. The devices were printed on technical paper (PEL P60) substrate (Printed Electronics Ltd). This substrate ensures optimal printability as compared to standard (i.e. for office use) paper. The printed photodetectors were dried at 80 °C under vacuum to evaporate the residual solvent.

Table 1. Simulation results for printed graphene NFC tag with different geometrical parameters.

2.2. Screen printed graphene electrodes and NFC

Graphite flakes (Alfa Aesar, 325 mesh, purity: 99.8%) were dispersed in 400 ml of N-Methyl-2-pyrrolidone (NMP) (Sigma, anhydrous, purity: 99.5%) in a concentration of 20 mg ml$^{-1}$ and shear mixed at 8000 rpm using Silverson L4R mixer equipped with square hole high shear screen for 2h at the constant temperature of 20 °C. The obtained dispersion then is transferred into glass bottle and bath ultra-sonicated (SHESTO, UT8061-EUK) for 24h. After that, the mixture is first filtered with 300-mesh stainless steel screen then the supernatant was carefully collected after centrifuged for 20 min at 500 rpm to remove any thick and unexfoliated flakes. The resulting dispersion is then vacuum filtered with Whatman filter paper grade 5 to remove the NMP. The graphene flakes collected on filter paper are re-dispersed in viscous solvent Ethylene Glycol (Alfa Aesar, anhydrous, purity: 99%) at concentration of 70 mg ml$^{-1}$ for screen printing. Screen printing was performed using semiautomatic screen printer with a 70° angle squeegee, at a printing speed of 50–150 mm s$^{-1}$. The printing screen with 24 mesh was fabricated and patterned with capillary film (ULANO, EZ50-Orange) to achieve the resolution and the adequate ink deposition for the printed circuit/ electrodes. Normal printing papers (Xerox Colotech A4 Paper 250 gsm) were used as substrate. The printed graphene NFC on paper is dried at 160 °C for 4h after printing and a further rolling compression is applied to enhance the printed film conductivity.

3. Results and discussions

There are two ways to connect a sensor to the RFID tag: the first method consists in printing the sensor directly on the RFID tag antenna, while the second method consists in connecting the two devices through an analogue-to-digital converter (ADC).

We started by investigating the first approach: a large size WS$_2$ photodetector was printed with active area of 1.5 mm $\times$ 1.5 mm, and was integrated with a screen-printed graphene meander line, figure 1(a). As the roughness of the screen printed graphene electrode may form pinholes in WS$_2$ layer and cause shorting, a thick WS$_2$ layer (thickness > 500 nm) was printed. The thickness of the WS$_2$ layer cannot be measured accurately due to the roughness of the paper substrate. The top electrode was inkjet-printed to touch the second screen-printed graphene electrode, so that a bias could be applied via the screen printed structure. The meander line structure was used for better experimental observation and alignment test. Although the $I$–$V$ measurements show relatively good photocurrent (figure 1(c)), a slow response time was observed. Thus this approach was aborted, and the second approach was investigated and adopted, i.e. the printed photodetector was connected to the chip on the printed graphene NFC tag antenna, which has the ability to read out values of the connected resistive component accurately from embedded ADC converter and transmit the data wirelessly through near field communication to the reader. The chip was powered wirelessly through drawing power from the magnetite field emitted by the NFC reader.
Figure 2. (a) Schematic of an all-printed heterostructure photodetector on a paper substrate, showing the graphene top (GrT) and bottom (GrB) electrodes and the WS$_2$ active layer in between. (b) Left: optical image of the device printed onto PEL P60 paper. Right: magnified optical image of the device corresponding to the red square in left mage.

Figure 3. $I$–$V$ curves of the photodetectors with increasing laser source ($\lambda = 532$ nm) power density made with 40 (a), 50 (b) and 60 (c) print passes of WS$_2$. (d) Photocurrent measured at $V_b = 2.5$ V as a function of increasing laser power density. The lines show the linear fit of the data, for each device.
A parametric study on the NFC tag antenna geometries has been carried out numerically in order to design the antenna with best possible performance. The printed graphene NFC tag antennas were simulated with different geometrical parameters: number of turns $N$, width of the coil $W$ and spacing between the adjacent coils $S$. The inductance $L_{\text{ant}}$ and resistance $R_{\text{ant}}$ can be obtained from the simulation. The quality factor of the antenna can be calculated from the resistance and inductance [29]. CST Microwave Studios was used for the NFC tag antenna simulation [30]. The printed graphene conductor was modelled as ohmic sheet with $1.5 \, \Omega \, \text{sq}^{-1}$. Simulated performance of the antennas with different structures is summarized for comparisons in table 1. The size of the final NFC tag antenna is $74 \, \text{mm} \times 53 \, \text{mm}$, fits in the credit card size limitation under ID-1 of ISO/IEC 7810 standard. For screen printed graphene antennas, a wide conductor width helps to reduce the ohmic loss. Because of the limited tag size, the tag antenna with more turns of coil is unable to have a wide conductor width $W$, such as 4 turns and 5 turns tag antennas, causing high resistance which hinders the performance. For this reason it can be observed from simulation that even with higher inductance from the 4 turns and 5 turns tag antennas, the antenna that gives the best performance is 3 turns one with width $W = 5 \, \text{mm}$, spacing $S = 0.5 \, \text{mm}$ and quality factor of 0.752. This design pattern is used in the screen patterning for screen-printing. The fabricated screen printed NFC tag antenna prototype was then connected to a calibrated vector network analyzer (VNA, Fieldfox N9918A, Keysight) using a SMA connector and characterized in polar mode to measure the tag antenna impedance. The resistance is obtained as $71.2 \, \Omega$ and the inductance is $0.54 \, \mu\text{H}$ at $13.56 \, \text{MHz}$. The corresponding quality factor is calculated as 0.65, slightly lower than simulation of 0.752 but the NFC tag has been verified experimentally to still be able to function properly. This is caused by unflatness of the exposed screen resulting in variations in sheet resistance of the printed antenna. The WS$_2$ photodetector is connected to the specially designed tiny PCB which holds the conversion circuit and functional NFC chip (RF430FRL152H; Texas Instruments). The PCB is then connected to the screen printed NFC tag antenna using conductive epoxy (CW2400, Circuitworks).

We have printed WS$_2$ photodetectors with 40, 50 and 60 print passes. Figure 2(a) shows the structure of the heterostructured photodetector. Three photodetectors were printed on the same piece of paper under exactly the same conditions. Figure 2(b) shows an optical micrograph of one of the photodetector, where the interface between the different layers can be clearly seen. The $I$–$V$ characteristics, in dark and under illumination, of the WS$_2$ photodetector (active area $2.25 \, \text{mm}^2$) were taken using a laser source (532 nm) of 3 mm beam diameter. The laser power is calibrated with optical power meter ML910A. Keithley 4200-SCS Semiconductor Characterization System is connected to the graphene electrode, while sweeping the voltage and recording the current. The current flowing across the samples with a sweeping voltage from $-2.5 \, \text{V}$ to $2.5 \, \text{V}$ under constant laser power densities of 0 mW cm$^{-2}$ (dark conditions), 44.1 mW cm$^{-2}$, 90.1 mW cm$^{-2}$, 128.0 mW cm$^{-2}$, 172.6 mW cm$^{-2}$ have been measured, respectively. As shown in figures 3(a)–(c), the $I$–$V$
curves of all three photodetectors are symmetric with non-Ohmic behavior where the resistance is observed to decrease with increasing laser power densities. The non-linear or non-ohmic IV curve confirms that multi-stacking layers were formed with no shunting pinholes within the photo-active WS$_2$ layer. Note that, the photodetector devices made with less than 40 print passes (e.g. 30 print passes) of WS$_2$ showed linear $I$–$V$ curves without any significant changes with increasing laser power illumination. This is due to the presence of pinholes in the WS$_2$ layer which causes the shunting of the devices. The non-linear or non-ohmic IV curve confirms that multi-stacking layers in our devices were formed with no shunting pinholes within the photo-active WS$_2$ layer. Nevertheless, figure 3(d) shows the photocurrent measured at bias voltage $V_b = 2.5$ V as a function of increasing laser power. The photo-responsivity of the printed photodetectors were 0.61, 0.46 and 0.41 mA W$^{-1}$ for devices with 40, 50 and 60 print passes of WS$_2$, respectively.

The photoresponses (change of resistance) of the integrated wireless printed graphene/WS$_2$ photosensors for different WS$_2$ samples under different laser power densities have been measured wirelessly using the measurement setup in figure 4 and the results are shown in figure 5. The same laser source for $I$–$V$ characteristic measurement in figure 1 was used whilst the reading is wirelessly transmitted to NFC reader (TRF7970A, Texas Instruments) and read out by a PC. The resistance change is normalized to 1090 $\Omega$, 3900 $\Omega$, 5900 $\Omega$ ($\pm 2\%$) respectively for 40, 50 and 60 printing passes. It is shown that the response for 50 and 60 printing passes with different laser power density are similar and the response for 40 passes is lower.

The real time photoresponse of the wireless printed graphene/WS$_2$ photosensor for a pulsed incident laser signal has also been measured for the sample made with 40 and 60 printing passes, as shown in figure 6. Each on/off state of the laser was set for approximately 60 s, laser power density is 172.6 mW cm$^{-2}$ for the on state. It can be seen that good repeatability is achieved for both samples during total measurement time of 300 s. The resistance increases as the thickness of the semiconducting layer (which domi-
nates the total resistance) increases. A video clip of a functional prototype is provided in the supplementary information (stacks.iop.org/TDM/7/024004/mmedia). The large change in resistance in on/off conditions indicates good responses to laser illumination, confirming instantaneous photodetection and successful wireless data transmission.

4. Conclusions

In this work, we have developed printed graphene/WS$_2$ enabled functional RF electronic device by combining large scale industrial screen-printing technology for large size and high conductive antennas and high resolution inkjet printing for more dedicated and smaller size heterostructured sensing devices. In addition, both antennas and sensing devices were printed on paper substrate, which is relatively low cost, biodegradable and disposable. Inkjet printed WS$_2$ photodetector was successfully integrated with screen printed graphene NFC tag antennas for wireless light sensing applications were demonstrated experimentally, revealing the potential of combining different printing technologies for development of printed 2D material-based devices for wireless sensing and IoT applications, compatible with low cost and flexible paper substrate. Inkjet printed sensing electronic components, along with reliable, inexpensive and large-scale screen printed circuit have a promising future in taking paper printed electronics to the next level without worrying compromising performance or substantially increasing the production cost for RF electronics.

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ORCID iDs

Ting Leng https://orcid.org/0000-0001-6559-6974
Daryl McManus https://orcid.org/0000-0002-8494-0870
Cinzia Casiraghi https://orcid.org/0000-0001-7185-0377

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