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*Published in:*

Proceedings of the 2021 13th Spanish Conference on Electron Devices (CDE)

*DOI:*

[10.1109/CDE52135.2021.9455738](https://doi.org/10.1109/CDE52135.2021.9455738)

Published: 25/06/2021

*Document Version*

Peer reviewed version

[Link to publication on the UWS Academic Portal](#)

*Citation for published version (APA):*

Douglas, C. I., Garcia Nuñez, C., Gibson, D., & Caffio, M. (2021). Development of flexible and high sensitivity graphene foam based pressure sensors. In *Proceedings of the 2021 13th Spanish Conference on Electron Devices (CDE): Sevilla, Spain 9-11 June 2021* (pp. 141-144). [9455738] (IEEE Conference Proceedings). IEEE. <https://doi.org/10.1109/CDE52135.2021.9455738>

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# Development of Flexible and High Sensitivity Graphene Foam Based Pressure Sensors

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**Abstract**— Pressure sensors are widely used devices in a variety of sectors from automotive, medical, industrial and consumer devices. These applications can range from ultra-sensitive e-skin, touch screen displays, medical diagnostics and health monitoring [1]. To compete with current industrial pressure sensors, a new easily fabricated, reproducible, and highly sensitive pressure sensor compatible with temperature sensitive substrates (plastic, fabrics, paper etc) is required. Within this paper the fabrication process is described, as well as showing the use of PDMS as a protective layer. Characterization of the fabricated sensors showed a sensitivity of 0.0418 mV/kPa over a range from 1 to 50 kPa. Cyclic testing showed that the use of a protective PDMS coating increased the durability of the sensors, keeping the voltage produced steady with no visible drop after large numbers of presses.

## I. INTRODUCTION

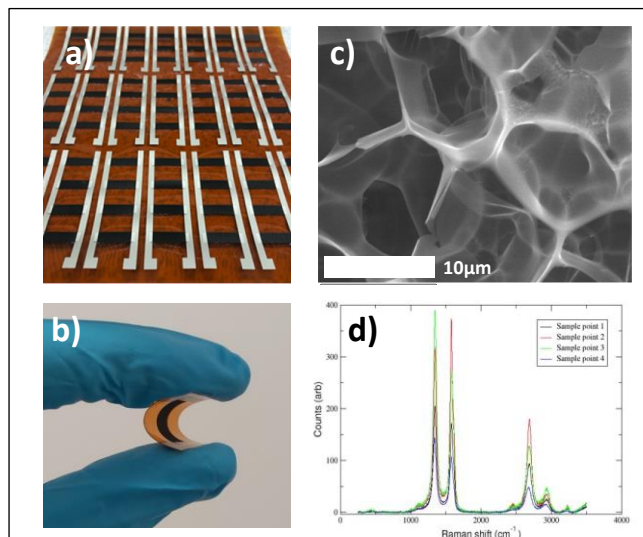
Currently flexible pressure sensors are primarily based on capacitive, resistive and piezoelectric methods. Capacitive presents advantages such as the ability to operate with no power supply. However, drawbacks include non-linear output and sensitivity to the surrounding environment. Piezoelectric sensors are durable and self-powering. Resistive pressure sensors provide advantages over the latter designs having simpler construction, robustness and long-term stability. Advanced materials such as graphene have been proposed as a promising material due to enhanced electric properties (fast device response, low recovery time and low power consumption), mechanical properties (high durability, conformability) providing compatibility with flexible substrates. Graphene foam (GF) is a three-dimensional (3D) high surface area nanostructure exhibiting excellent potential for development of pressure sensors. Currently graphene based resistive pressure sensors have shown high sensitivity at low pressures (<5kPa) enabling ultrasensitive detection [2]. It has also been shown that the use of graphene electrodes for capacitive pressure sensors provides a high sensitivity of 3.19kPa<sup>-1</sup> [3]. In this work, we show the growth of GF by a catalyst free method onto a flexible substrate without the use of a transfer process (Integrated Graphene Ltd proprietary process), and a novel structure consisting of GF embedded in polydimethylsiloxane (PDMS) used as an active layer in resistive pressure sensors. GF were directly grown on polyimide substrates (supplier Integrated Graphene Ltd,

Figure 1(a)). Morphology of the samples was characterized using SEM (Figure 1(c)). Resulting nanostructure structures, as well as the analysis of the defects density were characterized using Raman spectroscopy figure 1 (d).

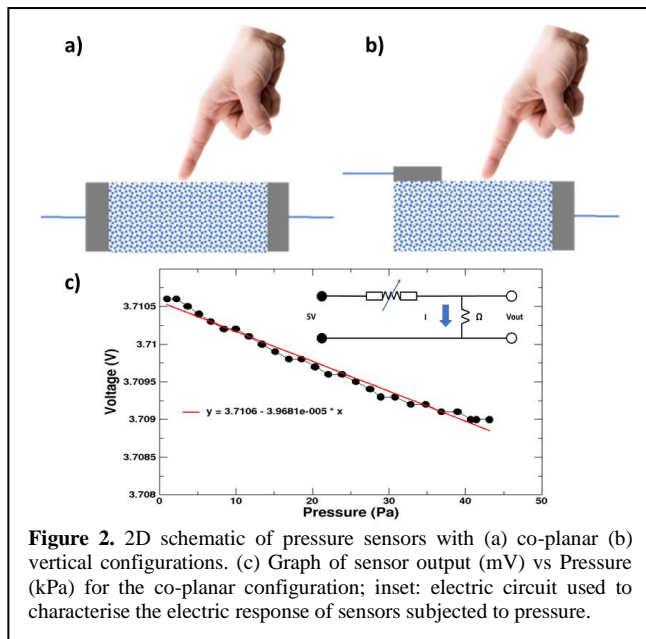
## II. EXPERIMENTAL DETAILS

Pressure sensors were fabricated with two configurations: i) co-planar and ii) vertical structure (Figure 2(a,b)). PDMS was subsequently embedded in the GF structure to enhance the sensitivity, increase the response time and provide enhanced robustness to resulting sensors. The first co-planar pressure sensor design was created by cutting out a slip of GF with Ag printed electrodes at each side, as shown in figure 1a. This was then placed on top of double-sided tape and positioned on top of a piece of PCB board. The PCB board was used to create electrical contact between the wiring and the Ag electrodes.

Once positioned on the PCB board wires were then soldered onto the board whilst using silver conductive epoxy (RS components (RS-186-3600)), to connect the wires from the Ag inkjet-printed electrodes to the PCB board. To provide



**Figure 1.** (a) Array of GF samples grown on polyimide substrates. Ag inkjet printed electrodes can be seen at each end of the GF pattern. (b) GF sample deformed by bending, showing flexibility and durability. (c) SEM image of GF, showing porous structure providing the high sensitivity of the pressure sensors. (d) Raman spectra of the GF measured at four different points (mapping).



mechanical and electrical properties of this silver conductive epoxy all solvents must be completely removed. This was achieved in one of two ways, leaving for 24 hours, or using a hot plate and baking between 121°C -> 148°C for 5 to 10 minutes. Once all solvent has been completely removed the sensor is ready for use.

Two types of co-planar sensors were created, with and without PDMS - formation of PDMS is explained later. The second design, vertical structure, was created by again cutting out a slip of GF however with complete removal of the Ag electrodes. A glass slide with a strip of double-sided tape has the GF sheet placed on top of the double-sided tape with the graphene side down. Using a small metal roller, with minimal pressure to avoid damaging the structure of the GF, the GF was rolled onto the double-sided tape to aid in adhesion. The Kapton substrate on which the GF was grown on was then peeled back, using a high peeling angle of >120°, leaving a strip of graphene present on the double-sided tape. Copper tape was then cut and placed at the ends of the GF to create electrical contact and wires were then soldered, one at each end of both pieces of copper tape.

Again, two variations were created, one with PDMS and one without. PDMS using a mixture of DOWSIL™ 184 Silicone Elastomer Base + DOWSIL™ 184 Silicone Elastomer Curing Agent. A ratio of 10:1 (base: curing agent) was used, using a precision scale to accurately measure the mass of each substance. Each substance was poured into a clean cup and using a metal spoon was mixed thoroughly (about five minutes), creating a bubbly white milk like consistency. Once thoroughly mixed the cup was placed inside of a vacuum oven, at room temperature, and all air was evacuated from the chamber. By evacuating the air from the chamber, in turn extracted the air bubbles from the PDMS mixture. Subsequently the PDMS was poured on top of the sensors creating a protective layer over the GF. To gather a thin uniform layer, the samples were stuck to a grinder, and rotated at ≈3000 rpm for 10 -> 20 seconds. Once the samples have been coated and spun, the PDMS is then cured in the oven at a temperature of 100 -> 120°C for one hour.

To gather results of voltage vs pressure, a simple electronic circuit was used. Within the circuit, a load resistor

was used with a constant input voltage ( $V_{in}$ ) of 5V, and the voltage drop across this resistor was measured with increasing pressures on the GF sensor (circuit diagram shown in inset of figure 2 (c)). Before each sensor was used, the initial resistivity of the sensor was measured to match the load resistor to a similar resistance range.

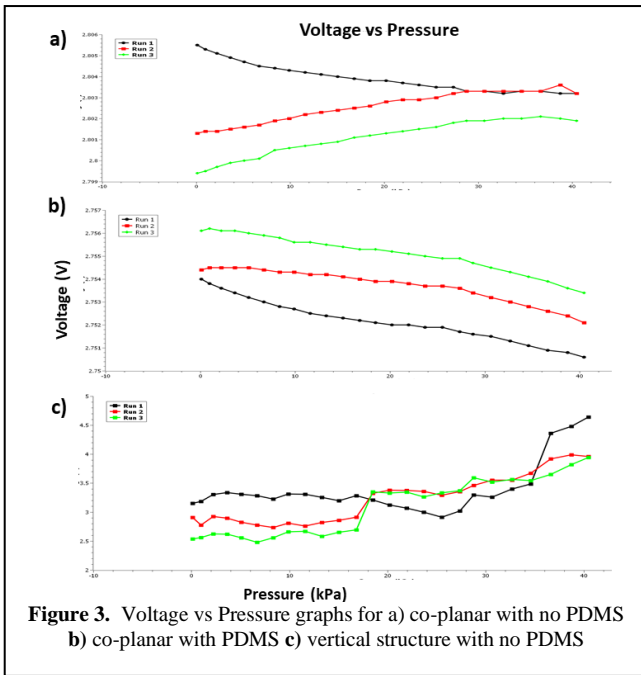
Characterization of the GF samples was performed using both Scanning electron microscopy (SEM) and Raman, (Figure 1 (c,d)). Raman analysis was performed on a DXR3(xi) Raman Imaging Microscope, using a 532nm laser. SEM was also conducted on the GF samples using a Hitachi S6400 cold cathode field emission SEM. Initially the GF was maintained on the Kapton sheet, however the images were not clear, therefore a transfer process was used to help improve image quality. The carbon discs used for SEM have a conductive “sticky” carbon side, which assisted the transfer process. The GF sheet was removed from the Kapton substrate by cutting the Ag electrodes off at each side and by placing graphene side down on the carbon disc. Again, using a high peeling angle, the Kapton was pulled back and the GF was transferred. This process greatly increased the image quality as shown in figure 1 (c). From the gathered SEM images, the structure of the three-dimensional graphene foam is clearly visible.

### III. RESULTS

To controllably pressure the samples, a programmable precision motor is used. This motor can move minimum distances of 0.1mm helping to accurately determine the amount of pressure being applied. To avoid damage to the sensor itself a mechanical finger was created using the same mixture of PDMS described above. To do this, the finger of a latex glove was cut and filled with PDMS, and a rod which attaches to the motor was placed inside the mixture which was then cured in the oven. After the initial design it was seen the finger was not directly vertical which would then give rise to inaccurate readings of pressure. To combat this a new design was created to hold the “finger” and another to hold the metal rod vertical. To calibrate the motor system, a weight balance (OHAUS Portable advanced – model no. CT1200-S) was used. The motor was moved in steps of 0.1mm, pressing down on the scale, and the mass on the scale was recorded. From this a relevant force could be determined. Using calipers, the area of the finger could be determined and from  $P=F/A$ , a pressure was calculated. From this calibration we can easily determine the pressure produced at any given distance.

As can be seen in figure 1 (d) the D peak as well as the G peak are both intense, positioned at 1350 $cm^{-1}$  and 1578 $cm^{-1}$ . The G band is due to the doubly degenerate  $E_{2g}$  mode at the Brillouin zone centre, whereas the D band arises from the defect mediated zone-edge phonons. The 2D band originates from the second order-double resonant raman scattering from zone boundary,  $K+\Delta K$  phonons [4].

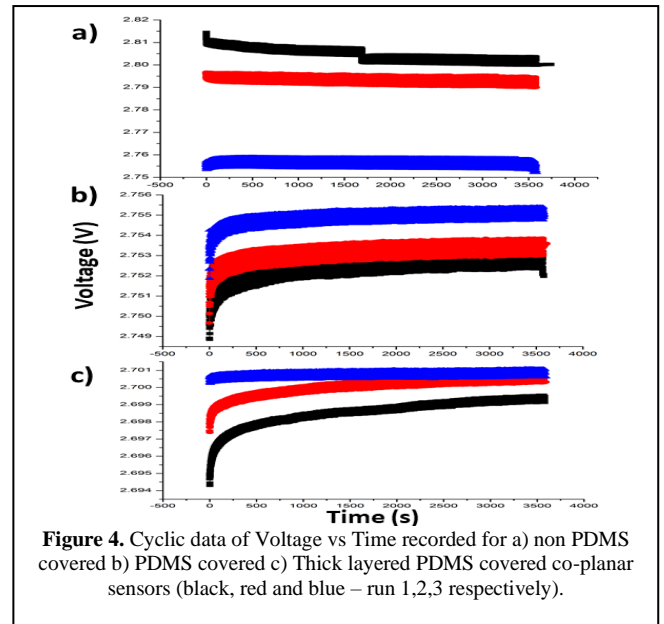
The D/G ratio provides an indication of the level of defects within the GF sheet, with higher D/G ratios showing a higher defect density. As can be seen from table 1, this ratio ranges from 0.9 to 1.3. The G peak, graphitic peak, provides an indication of the  $sp^2$  hybridization of the GF sheet. Results indicate high levels of  $sp^2$  hybridization due to the sharp intense G peak. The 2D peak, approximately 2685 $cm^{-1}$ ,



provides an indication of the number of layers of graphene. Looking at the 2D/G ratio, an indication of the layer number can be assumed, with lower numbers of the 2D/G ratio resulting in lower layer numbers [4]. As can be seen in table 1, the 2D/G ratio is approximately 0.85 indicating multilayer graphene. A ratio of  $\sim 0.85$  indicates approximately 6 layers of graphene. This was expected due to the fact it is a 3D graphene foam sheet and not single layer graphene. Original measurements were taken with both the vertical structure and the co-planar structure with a layer of PDMS on top. It was observed for the co-planar configuration that as the pressure was increased this led to a decrease in the voltage produced. For the vertical structure, the opposite was seen, with a voltage increase shown.

New prototypes were developed and both PDMS covered and non PDMS covered were then tested. Testing of the new co-planar sensors shows that, when covered in PDMS that the voltage decreased as the pressure increased whereas with no PDMS layer the first test showed a decrease in voltage but tests after that show an increase. Figure 3 shows the respective graphs for the new batch where it can be seen there is a clear change in the characteristics of the sensor. For the vertical structure with no PDMS, it could clearly be seen that there is an increase in the voltage produced.

The sensors themselves produce an unstable voltage, where the initial starting voltage (with no pressure applied) changes from 3.17V to 2.9V to 2.5V leading to it being not possible to gather results when covered with PDMS. For the co-planar sensors, the change in voltage is stable, within the millivolts range, however for the vertical structure the change is unstable causing most results gathered to have a rather large error caused due to large unstable fluctuations in the 0.1V range. Due to this fact it was more beneficial to concentrate on the co-planar configuration whilst trying to develop a new design for the vertical structure which may increase the stability of the sensor itself.



a set number of times, in this case 1000 repetitions. Testing of the durability was conducted on PDMS and non PDMS covered co-planar sensors, figure 4 (a,b). It can be seen in figure 4 that for the non PDMS covered sensors the voltage over time tends to decrease and plateau, with the first test including a major drop in voltage.

This drop could have been caused by many factors i.e., a slight decrease in height of the sensor stage or slight movement of the sensor wiring, however could also be caused by a break in the GF structure itself. For the PDMS covered sensors, the voltage increases and plateaus again. This shows good durability of the sensors as there is no visible sign of voltage drop or a sharp increase at large numbers of presses. To test if the thickness of PDMS made an impact on the durability, another sensor was created with a thick layer of PDMS present. This sensor was not put on the grinder so that the PDMS did not spread. Again, from this you can see that there is a clear increase in voltage following the PDMS covered characteristics, figure 4(c).

#### IV. CONCLUSIONS

GF porous structure is observed from SEM imaging (Figure 1(c)). Further analysis determines action of the GF, particularly when voids in the GF are compressed closer together and relaxed. From applying a constant input voltage ( $V_{in}$ ) of 5 V and using a load resistor with a value similar to the resistance of GF (i.e.  $R_{load}=25\Omega$ ) the  $V_{out}$  was observed to decrease with the applied pressure (Figure 2(c)), indicating that the resistance of GF ( $R_{GF}$ ) increases with pressure. From this result, it is postulated that the voids size increased with the pressure due to the expansion of the PDMS layer integrated in GF. PDMS layer presented various roles, comprising protection to GF, faster recovery times, and more sensitivity to a wider range of pressures compared to bare GF. Characterization showed a sensitivity of 0.0418 mV/kPa over a range of pressures from 1 to 50 kPa. Cyclic testing proved that the PDMS layer does increase protection of the GF itself. This could be shown by the drop in voltage after each test run with no PDMS present, going from 2.81V to 2.79V to 2.75V. Whereas for the PDMS covered sensors the voltage stays

Analysis Position	D Peak (cm <sup>-1</sup> )	G Peak (cm <sup>-1</sup> )	2D Peak (cm <sup>-1</sup> )	D/G ratio	2D/G ratio
#1	1354	1571	2685	1.166	0.918
#2	1357	1578	2688	0.974	0.834
#3	1356	1581	2686	1.351	0.845
#4	1342	1576	2675	1.327	0.837

**Table 1.** Raman data showing the mapping sample points and relevant peak position, D/G and 2D/G ratios.

steady with no visible drop. To gather more precise data for the vertical structure a new design must be created. This is vital as the sensors cannot be used in the unstable state they currently produce. As the sensors are still in the initial fabrication process more data needs to be collected and analyzed to fully understand the implications these sensors can provide for a variety of industries.

#### ACKNOWLEDGMENT

I would like to take this time to thank my academic and industrial supervisors for all the time spent with myself working on this project. I would also like to thank the technicians at the University of the West of Scotland Institute

of Thin Films, Sensors and Imaging for their help on the project. This research was partially funded by Integrated Graphene Ltd, University of the West of Scotland and SRPe.

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